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(OPERATION OF A FORCED CIRCULATION, HAYNES ALLOY NO. 25, MERCURY LOOP TO STUDY CORROSION PRODUCT SEPARATION TECHNIQUES)

by David B. Cooper

Prepared under Contract No. NAS 5-462 *by*
THOMPSON RAMO WOOLDRIDGE, INC.
Cleveland, Ohio
for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JULY 1965

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FOREWORD

This report is concerned with the construction, operation, and evaluation of a forced circulation mercury loop designed to investigate corrosion product separation techniques in a Haynes alloy No. 25 system. The work was performed between May 1962 and April 1964 in support of the Sunflower program under NASA Contract NAS 5-462 with Mr. Jack A. Heller, Space Power Systems Division, NASA Lewis Research Center, as Technical Manager. The report was originally prepared as Thompson Ramo Wooldridge Report ER-5985, May 1, 1964.

OPERATION OF A FORCED CIRCULATION, HAYNES ALLOY NO. 25,

MERCURY LOOP TO STUDY CORROSION PRODUCT

SEPARATION TECHNIQUES

by David B. Cooper

Thompson Ramo Wooldridge Inc.

SUMMARY

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A Haynes alloy No. 25, forced circulation, mercury loop was designed and operated for a total of 5218 hours at an average boiling temperature of 1097°F. Corrosion product separators were included in both the vapor and liquid sections of the system and were evaluated for their effectiveness in reducing problems associated with mass transfer in mercury systems. Corrosion data for this system were found to agree favorably with previously reported data for two-phase, thermal convection loops. The greatest attack was found in the low vapor quality regions of the boiling section, one isolated portion of the superheater inlet, and in the orifice and nozzle where condensation was occurring. The condenser and subcooler sections of the loop suffered negligible attack, as did the dry portions of the superheater. Deposition was observed in the loop and was greatest in the preheater section and in the orifice and nozzle where condensation was occurring. The action of the corrosion product separator located in the post-condenser region of the loop prevented deposition in the subcooler section. The vapor corrosion product separator was approximately 51 percent efficient in removing corrosion products carried over from the boiler. Operation of this loop for 5218 hours has demonstrated the feasibility of long-term operation of a Haynes alloy No. 25 mercury system.

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OPERATION OF A FORCED CIRCULATION, HAYNES ALLOY NO. 25, MERCURY LOOP TO STUDY CORROSION PRODUCT SEPARATION TECHNIQUES

by David B. Cooper

Thompson Ramo Wooldridge Inc.
Cleveland, Ohio

INTRODUCTION

The materials problems associated with the design and operation of a mercury Rankine cycle power system are fairly well defined. With the exception of refractory metals, no known material is entirely resistant to corrosive attack by mercury at high temperatures. While the loss in strength of the containment material due to the attack by mercury is generally not great enough to reduce the durability of the material significantly, the generation of corrosion products results in a number of secondary problems which seriously threaten the long-term reliability of the system. These problems center around the transfer of the corrosion products from one portion of the system to another, with the resultant effect that the efficiency of the system may be reduced. More serious than this is the fact that regions such as the turbine may become plugged with corrosion products. One solution to the problem is that of continuous separation of the corrosion products from the working fluid by such techniques as chemical gettering and the use of magnetic fields. Corrosion product separation work at TRW on thermal convection loop systems with flow rates of 10 to 40 pounds per hour has shown that as much as 85 percent of the corrosion products generated can be removed in such a loop (1).

In a system such as Sunflower, continuous separation of the corrosion products from the working fluid appears to be the most feasible method for avoiding mass transfer difficulties. In order to evaluate the effectiveness of corrosion product separators, a forced circulation mercury loop was designed, constructed, and operated. The loop was designed for long-term (5000 hours) operation at conditions simulating those found in the Sunflower system and included two corrosion product separators. Evaluation was concerned with corrosion and mass transfer effects as well as the effectiveness of the corrosion product separation techniques employed.

SYSTEM DESCRIPTION

A. Loop

The design parameters for the corrosion product separation loop were chosen so as to simulate the anticipated conditions in the final Sunflower system, with the exception of flow rate. These conditions included:

Boiler outlet temperature	1100°F
Superheater outlet temperature	1250-1300°F
Condensing temperature	650°F
Subcooler temperature	300°F
Flow rate	73 lbs./hr.

The design flow rate was approximately 1/12 of the flow rate for the Sunflower system.

Haynes alloy No. 25 was the basic material of construction and was chosen because of its relative resistance to mercury corrosion at temperatures up to 1000°F (2) and its strength at elevated temperatures. Type 316 SS was used in low temperature regions of the loop for instrument sensing units and valving. Several other materials were used in small quantities in some sections of the loop and will be discussed in the detailed description of the system which follows. Figure 1 shows a schematic of the loop and is referenced during the following description.

1. Boiler and Superheater

The boiler and superheater sections of the loop were fabricated from 1.0 inch O.D. X 0.065 inch wall Haynes alloy No. 25 tubing. Electrical resistance-type heaters were used to supply energy to these sections and were designed to supply maximum overall heat fluxes of 15,000 and 10,000 Btu/hr-ft^2 in the boiler and superheater, respectively. Actual fluxes used during operation were in the order of 5,000 Btu/hr-ft^2 over eight feet of heated boiler tubing and 2100 Btu/hr-ft^2 over 5.75 feet of heated superheater tubing. The heaters were fabricated in place by wrapping Nichrome heater ribbon around refractory tubing which surrounded the loop tubing. A solution of a refractory cement in water was spread between the loop tubing and the refractory tubing to provide better heat transfer. The cement was also spread on the outside of the heater assembly to hold the Nichrome ribbon in place and to insulate the unit. In order to create turbulence and facilitate the heat transfer, swirl wire was used throughout the boiler and superheater. Haynes alloy No. 25 wire of 0.062 inch diameter was coiled into a helix having an outside diameter of 0.870 inch and a pitch of 3.5 inches.

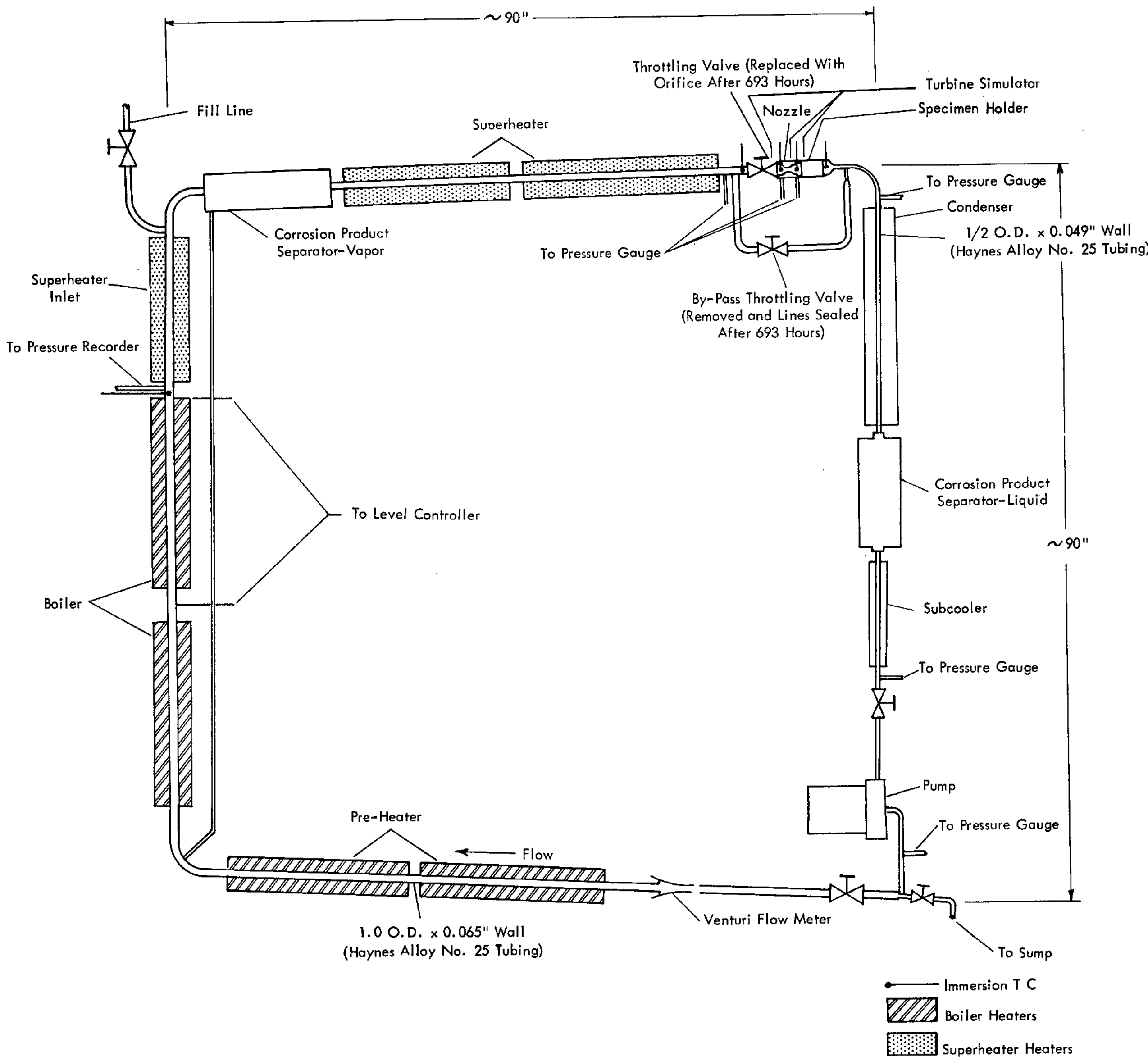

FIGURE 1 SCHEMATIC OF CORROSION PRODUCT SEPARATION LOOP

2. Condenser and Subcooler

The condenser and subcooler sections of the loop consisted of single Haynes alloy No. 25 tubes having an outside diameter of 0.5 inch and a wall of 0.049 inch. Cooling of these sections was accomplished primarily with two 195 CFM ventilating blowers. Both the condenser and subcooler tubes were enclosed within four-inch square by 12-inch long manifolds. In addition to the air blowers, 0.125 inch copper cooling coils were wrapped around the condenser and subcooler tubes to provide additional cooling capacity.

3. Throttling Valves and Orifice

A bellows-sealed, manually-operated, throttling valve was located between the superheater and condenser sections and provided the required pressure differential in the system. A by-pass line was installed around the throttling valve and contained an automatically-controlled throttling valve which provided the required pressure drop should the first valve fail. The two valves were identical with the exception of control and were fabricated entirely from Haynes alloy No. 25. After 693 hours of operation, a leak developed in the manually-operated valve, and this was replaced with a sharp-edged orifice. The automatically-controlled valve was also removed at this time since a leak test of the valve indicated a defective bellows. The by-pass line was not removed but was, instead, sealed with Haynes alloy No. 25 caps.

The orifice plate was fabricated from Haynes alloy No. 25 and was 0.032 inch thick. The orifice was 0.045 inch in diameter.

4. Nozzle and Erosion Specimen Holder

A nozzle was also used in the system and was located after the throttling valve (or orifice). Although the nozzle provided a small pressure drop, the primary purpose was to investigate the erosion resistance of Nivco 10*, which was the material of construction. The nozzle is shown in Figure 2.

Located immediately following the nozzle was a Haynes alloy No. 25 erosion specimen holder, shown in Figure 3. The erosion specimen holder contained four specimens (see Figure 4) and included:

- (1) PH15-7Mo (RH 950 heat treatment)
- (1) PH15-7Mo (TH 1050 heat treatment)
- (2) Nivco 10

These materials were chosen as being representative of the design material for the Sunflower turbine.

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*Nominal analysis:	C	0.02	Fe	0.30	Ti	1.80
	Mn	0.35	Al	0.22	Ni	22.50
	Si	0.15	Zr	1.10	Co	73.50

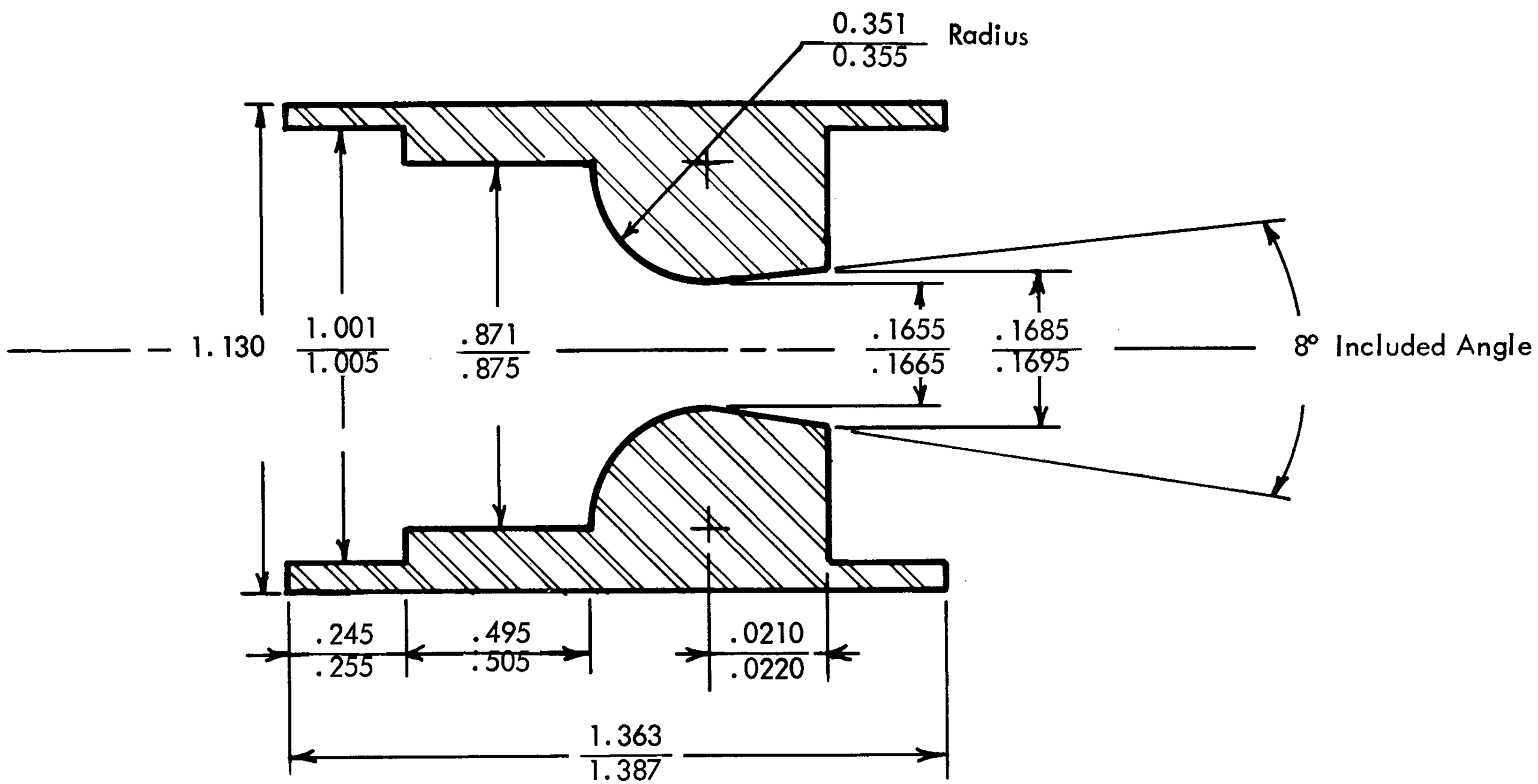

Figure 2. NIVCO 10 NOZZLE

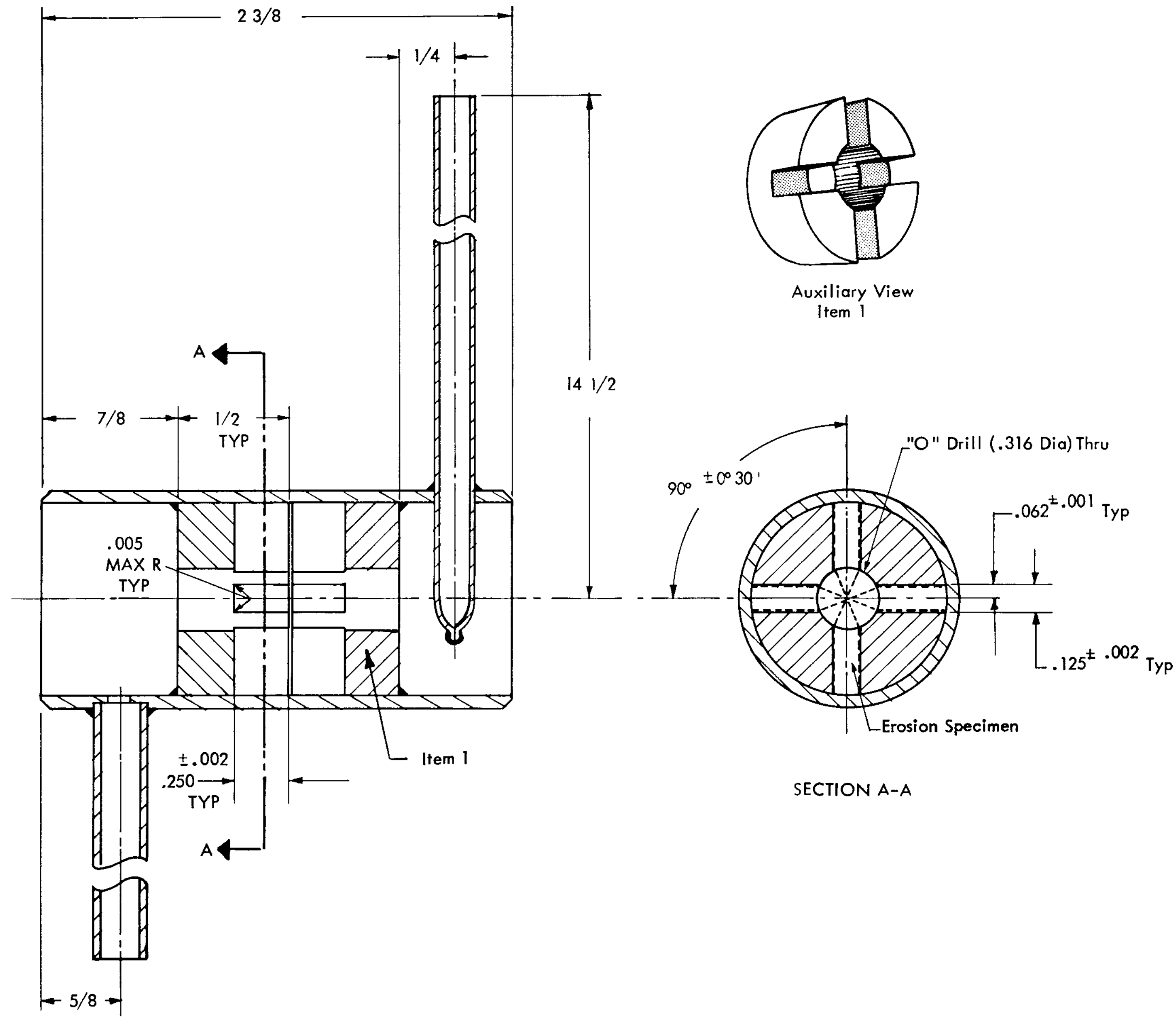

FIGURE 3 EROSION SPECIMEN HOLDER

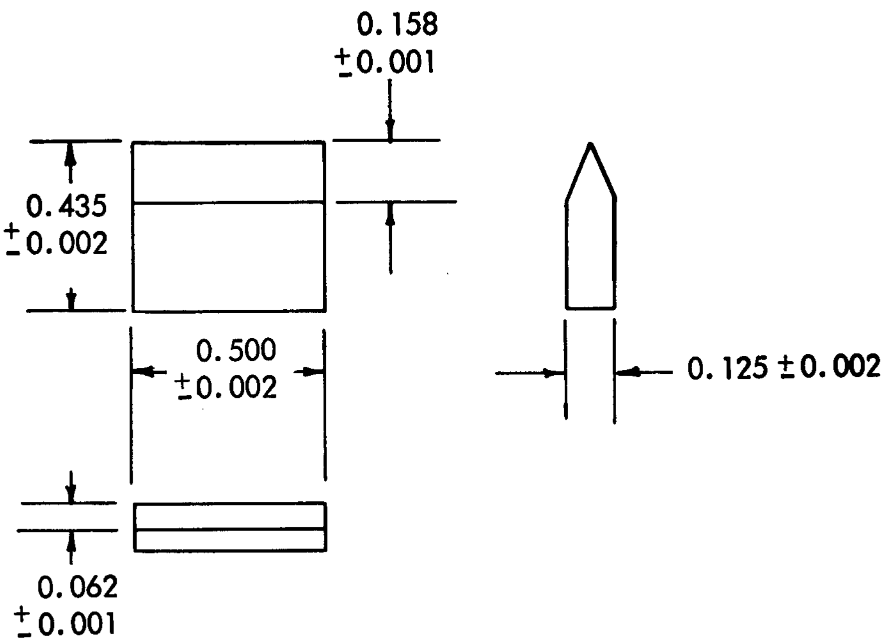

Figure 4. EROSION SPECIMENS

5. Corrosion Product Separators

Two corrosion product separators were included in the loop and are shown in Figures 5 through 8. The first of these, the vapor corrosion product separator, was located in the superheater section of the loop (see Figure 1). The separator utilized 0.875 pounds of columbium (E.I. duPont alloy D-36, containing 10 percent titanium and 5 percent zirconium) turnings as a getter for separation of corrosion products carried over in the vapor from the boiler. A drain line was included in the separator so that any separated liquid would be returned to the boiler. The separator was fabricated from Haynes alloy No. 25, with the exception of the columbium alloy getter.

The second separator was located at the condenser exit and utilized both magnetic and chemical gettering principles. The mercury exiting from the condenser passed first through 4.4 pounds of columbium (10% titanium, 5% zirconium) turnings and then through 1.6 pounds of low carbon steel wool. From this point the mercury passed upwards towards the separator entrance between the poles of three 3000-Gauss "C"-shaped Alnico 5 magnets. The magnets were not enclosed, but were in contact with the mercury. After passing through the magnets, the mercury then flowed through 1.6 pounds of low carbon steel wool and into the outlet tube to the subcooler. All parts of the separator were fabricated from Haynes alloy No. 25, with the exception of the getters, magnets, and Type 410 SS pole pieces.

B. Controls

1. Heater Controls

Power to the heaters on the boiler, superheater, and the vapor corrosion product separator sections of the loop was varied by the use of General Radio "Variac" autotransformers. Each heater circuit also contained a Weston ammeter. Alnor Type N-14 millivoltmeter-type temperature controllers were used to control the temperature of each heated section of the loop. Temperature indication was made by chromel-alumel thermocouples secured to the surface of the loop tubing near the exit of each heater circuit.

2. Condenser and Subcooler Control

The flow of cooling air for the condenser was varied by means of a Minneapolis-Honeywell motor-operated butterfly valve. The input signal to the motor was received from a Weston high-low, millivoltmeter-type temperature controller. This instrument sensed the condenser temperature as indicated by an iron-constantan thermocouple secured to the condenser wall.

The flow of cooling air for the subcooler was manually controlled by means of a butterfly valve. The cooling water for the condenser and subcooler sections was manually controlled.

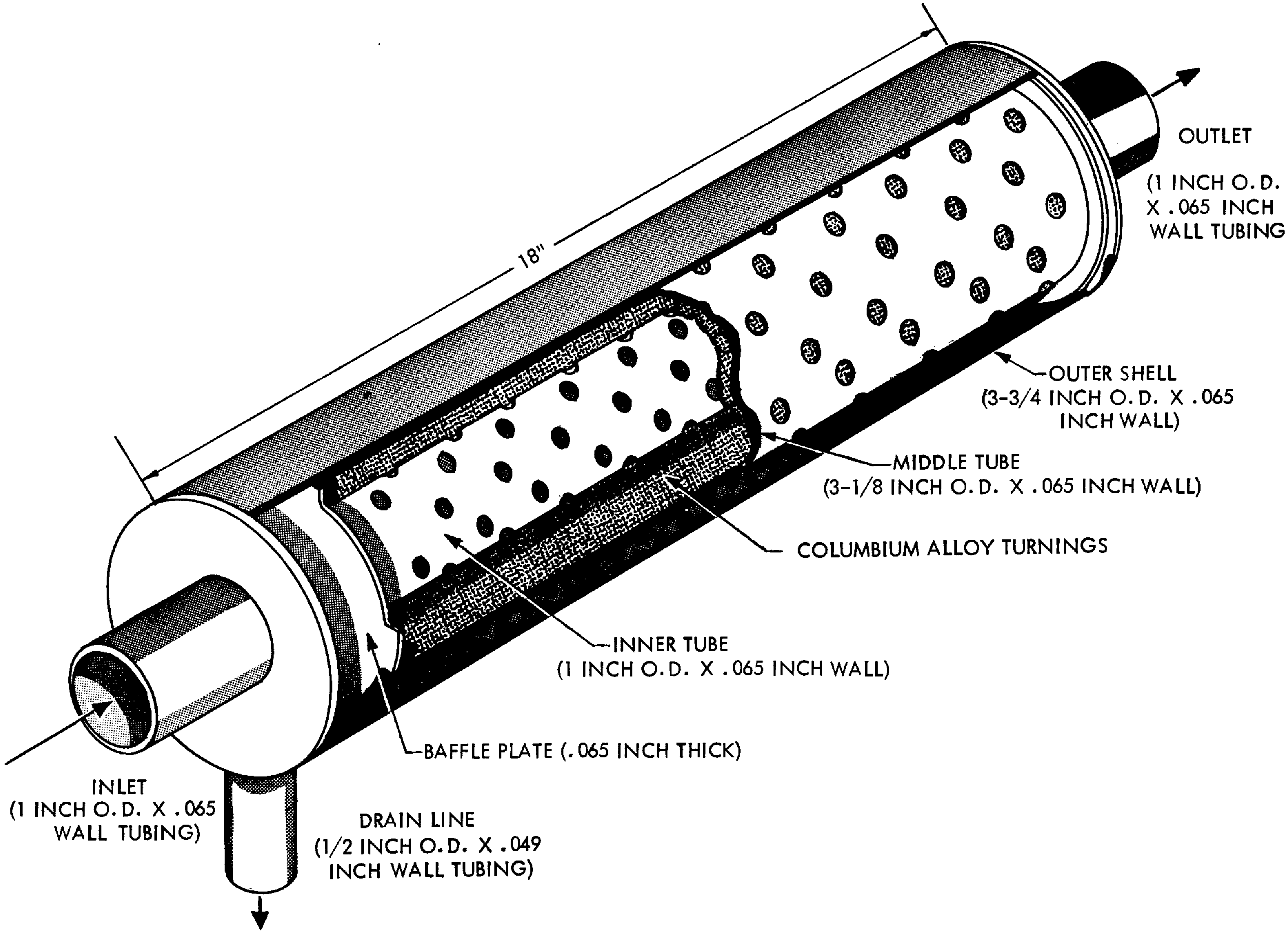

FIGURE 5. VAPOR CORROSION PRODUCT SEPARATOR.

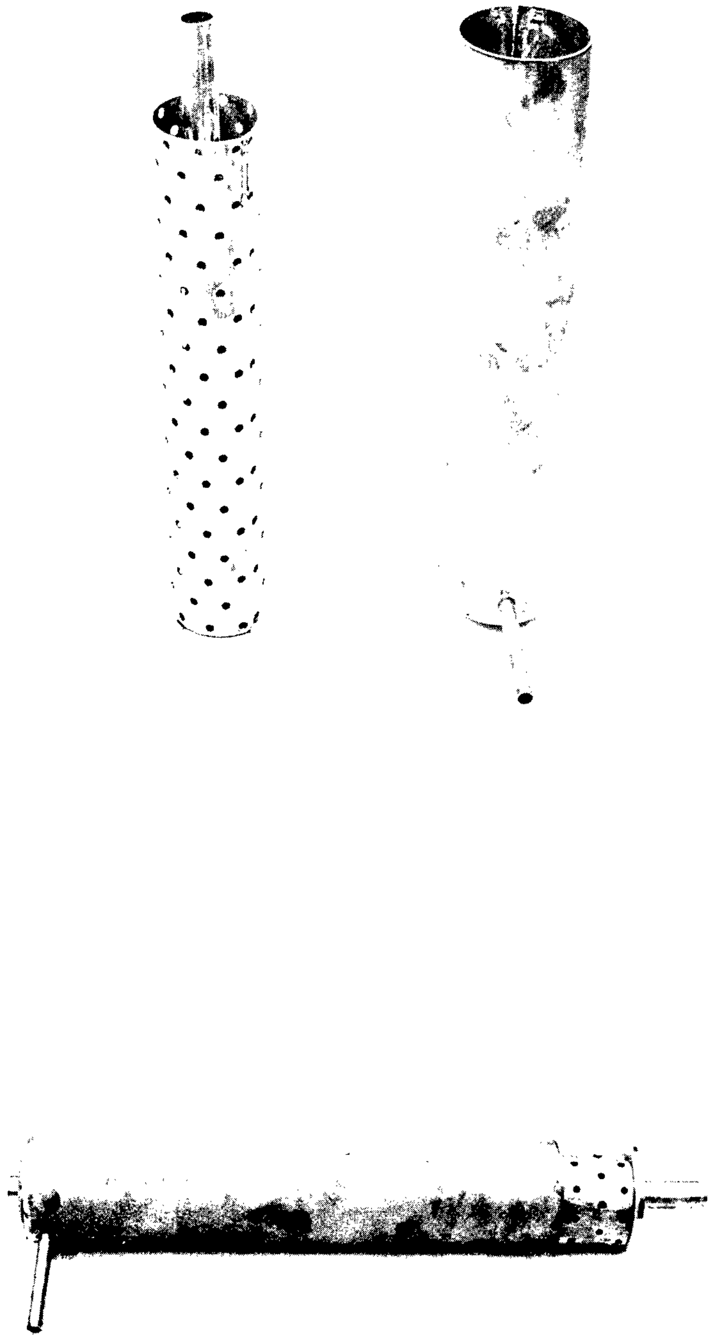

Figure 6. Vapor Corrosion Product Separator.

ITEM No.	REQD.	PART NO.	DESCRIPTION	[redacted], MAT'L
15	1		POLE PIECE, SHAPED	AISI-410 S.S.
13	1		POLE PIECE, SHAPED	AISI-410 S.S.
12	1		END PLATE, PERFORATED	HAYNES 25
11	1		OUTLET TUBE	HAYNES 25
10	A.R.		Cb TURNINGS (WOOL)	DENSITY APPROX. .015LBS/IN³
9	A.R.		Fe TURNINGS (WOOL)	DENSITY APPROX. .015LBS/IN³
8	1		RETAINING PLATE, MAGNET	HAYNES 25
7	1		INNER SHELL	HAYNES 25
6, 6A	1 EA.		OUTER SHELL	HAYNES 25
5	3		MAGNET	ALNICO 5
4	1		SEPARATING PLATE	HAYNES 25
3	1		END PLATE, PERFORATED	HAYNES 25
2	2		END PLATE	HAYNES 25
1	1		INLET TUBE	HAYNES 25

LIST OF PARTS

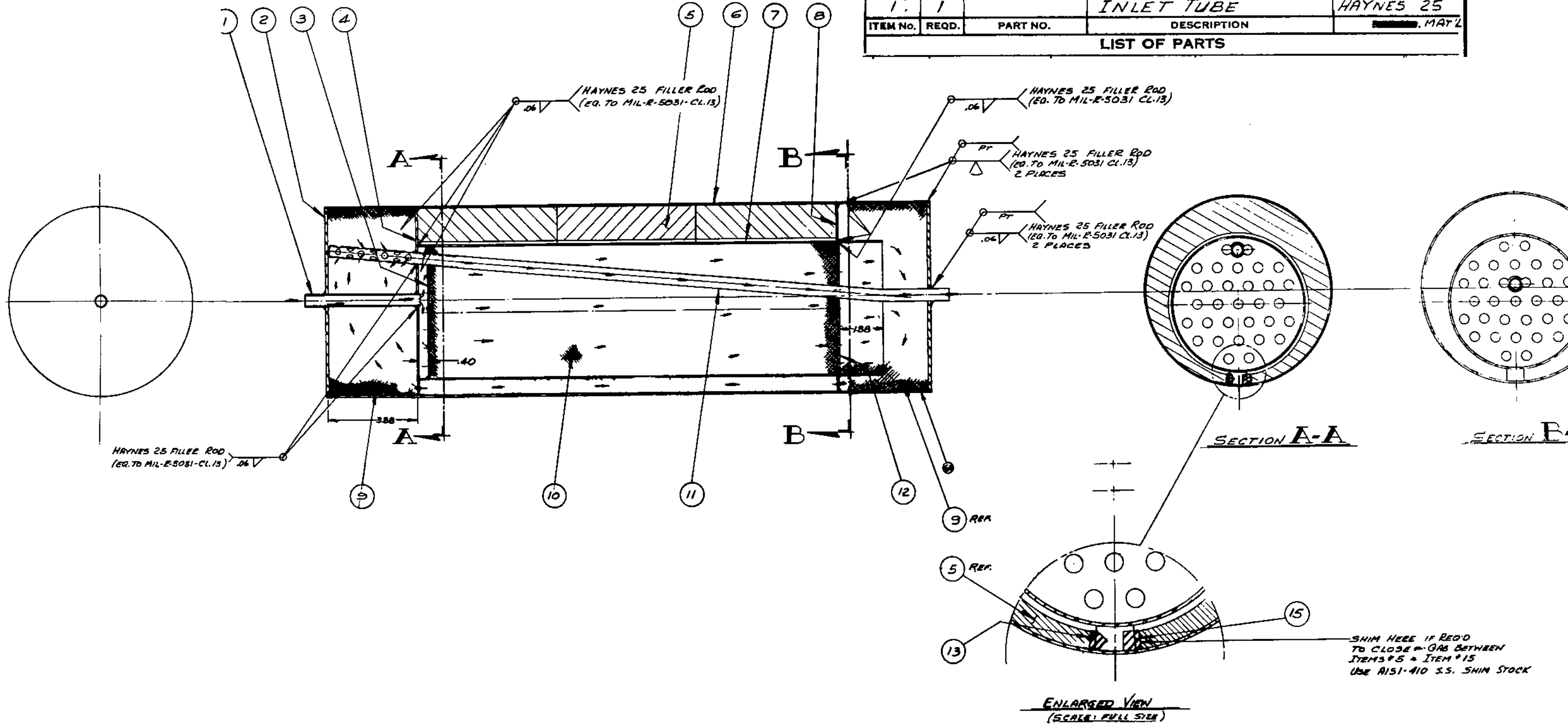

Figure 7. Liquid Corrosion Product Separator.

Figure 8. Liquid Corrosion Product Separator.

3. Pressure Measurement

A Minneapolis-Honeywell pressure recorder with an over-pressure control circuit provided a record of the boiler outlet pressure and also served as a safety device in the event of an over-pressure situation. A total of five Ashcroft pressure gauges and one Telco "Helicoid" gauge were used to measure the pressure at the various locations in the system listed below:

1) Superheater outlet pressure
2) Nozzle inlet pressure
3) Nozzle differential pressure
4) Condenser pressure
5) Pump inlet pressure
6) Pump outlet pressure

All instruments were actuated by Type 316 SS diaphragm seals connected to the loop by Haynes alloy No. 25 tubing. A constant head of liquid mercury was maintained in the sensing units, and all pressure readings have been corrected for this pressure head.

4. Temperature Measurement

In addition to the control thermocouples, forty-six others were placed at various locations around the loop. Five of these were immersion thermocouples and were located at the boiler exit, at the superheater exit, at the nozzle inlet and outlet, and at the specimen holder outlet. The remaining forty-one were secured to the surface of the tubing. All thermocouples were chromel-alumel and were recorded on a 48-point Weston temperature recorder which was equipped with a limit switch for protection of the loop against excessive temperature.

5. Pump and Liquid Level Control

The pump used in the loop was a Milton Roy positive displacement pump with a pneumatic operated U.S. "Varidrive" variable speed motor. Materials of construction in contact with the mercury included:

Liquid end	Type 316 SS
Diaphragm	Teflon
Ball seats	Type 316 SS
Ball valves	Type 416 SS
Check valve housing	Type 316 SS

The pump had a capacity of 158 pounds per hour of mercury (specific gravity of 13.6) and a maximum discharge pressure of 500 psig.

A Minneapolis-Honeywell level controller was employed to maintain the proper quantity of mercury in the boiler. Indication of the level was given by a Type 316 SS bellows assembly which sensed the difference in pressure at two points in the boiler section twenty-four inches apart. A pneumatic signal was transmitted by the level controller to the variable speed motor on the positive displacement pump.

A Type 316 SS Venturi having a 0.035 inch diameter orifice was used to measure the mercury flow at the boiler entrance. The pressure drop across the Venturi was indicated by an inverted U-tube manometer of Type 316 SS construction.

6. Throttling Valve By-Pass

Control of the throttling valve by-pass valve was achieved by means of an Ashcroft pneumatic transmitter containing a Type 316 SS Bourdon tube. The transmitter was calibrated such that a decrease in the condenser pressure to less than 10.5 psia would result in the sending of a pneumatic signal to the by-pass valve, allowing the valve to open. The transmitter would then control the condenser pressure between 7.5 and 10.5 psia by varying the setting of the by-pass valve.

C. Enclosure

The entire system, with the exception of the pump and instrument sensing units, was installed in a two-foot square enclosure fabricated from sheet metal and Unistrut framing supports. After installation of the loop, the enclosure was filled with vermiculite which served as the insulation. A photograph of the loop installed in the enclosure prior to sealing is shown in Figure 9.

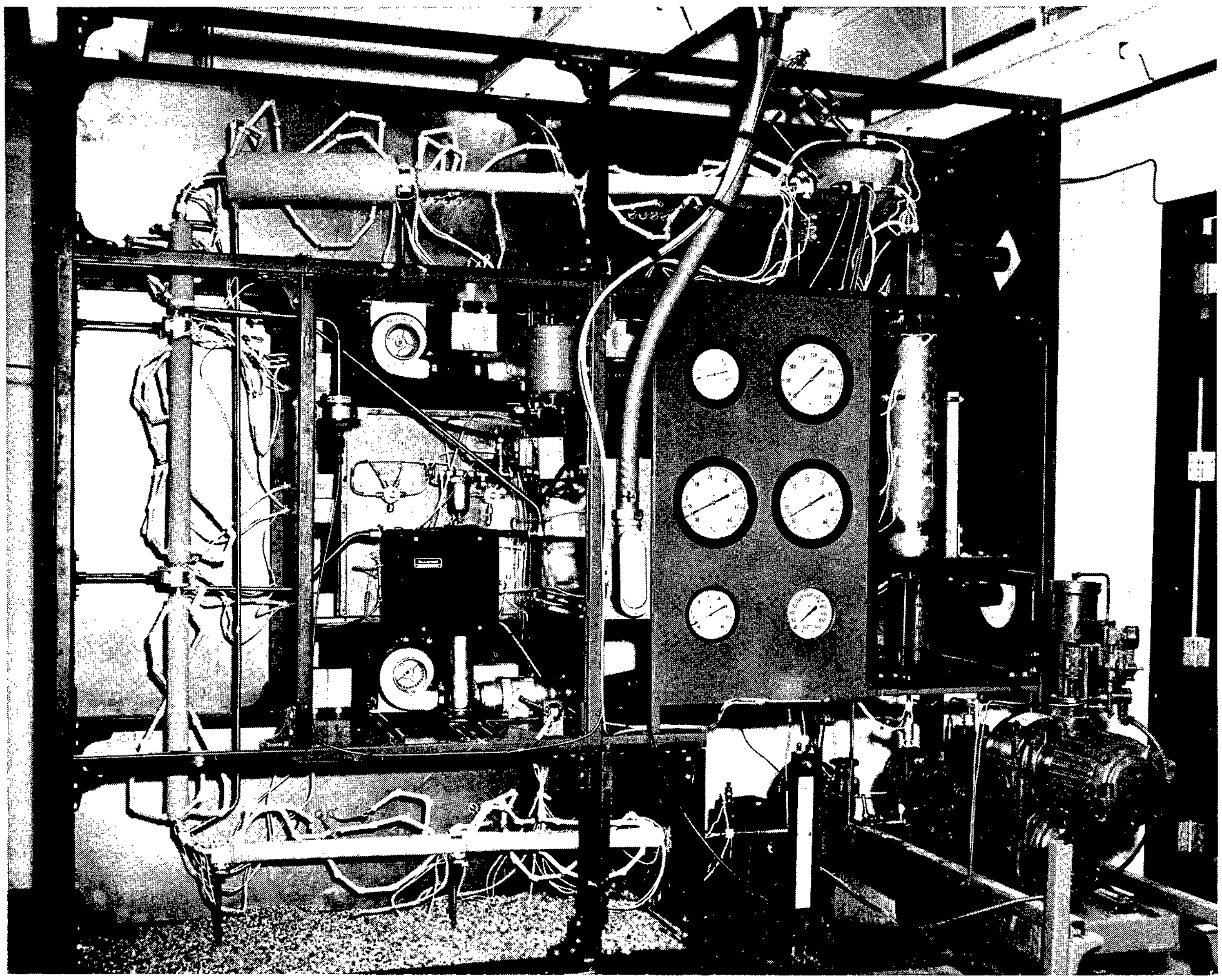

Figure 9. Corrosion Product Separation Loop.

FABRICATION, ASSEMBLY, AND OPERATION

A. Fabrication and Assembly

The various components of the loop were machined and/or formed using standard shop practices. Before assembly, all components except the one-inch Haynes alloy No. 25 tubing were immersed in a descaling salt bath* for two minutes at 850-900°F. Upon removal, the parts were rinsed with water and then immersed in a 10 percent sulfuric acid solution at 140°F, followed by a second water rinse. The parts were then dried with analytical reagent-grade acetone.

Prior to fabrication, the one-inch Haynes alloy No. 25 tubing was annealed at 2250°F in a hydrogen atmosphere with a dew point of -80°F. The tubing was held at this temperature for 15 minutes and was then furnace cooled. The various bending and machining operations were then performed on the tubing, followed by a second anneal at 2250°F in a dry hydrogen atmosphere. Before assembly, the tubing was rinsed with analytical reagent-grade acetone.

The loop components were installed in the loop enclosure and were welded in place by the tungsten inert-gas process using L-605 (Haynes alloy No. 25) filler rod. The system was then leak tested under both argon pressure and vacuum and was found to be leak-tight. The loop was then outgassed for a period of 70 hours, and a leak rate of 26 microns per hour was established.

B. Operation

The loop was filled with triple distilled, A.C.S.-grade mercury, and the system was pre-treated with liquid mercury for 90 hours at 600 to 700°F. The average flow rate during this flushing operation was 58 pounds per hour. Following the pre-treatment, the loop was drained except for the liquid corrosion product separator, re-filled with clean mercury, and test operation was begun.

During the course of the test operation, several failures occurred in the system and changes were made to correct these failures. Because of the nature of some of the modifications, loop conditions varied during different periods of operation. For this reason, the test operation has been divided into four runs which are discussed separately. Average operating conditions for each run are given in Table 1.

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*Virgo descaling salt obtained from Hooker Chemical Corp., Niagara Falls, N.Y.

TABLE 1

AVERAGE LOOP OPERATING CONDITIONS

		Thermocouple Number	Run No. 1	Run No. 2	Run No. 3	Run No. 4
Total operating time, hours			0-693	693-759	759-1013	1013-5218
Elapsed time, hours			693	66	254	4205
Thermocouple Locations and Average Reading, °F						
Boiler Heater No. 1 Inlet		2	758	759	797	838
Boiler Heater No. 1 Outlet		3	833	838	888	925
Boiler Heater No. 1 - Boiler Heater No. 2		4	897	953	991	1022
Boiler Heater No. 2 Inlet		5	947	1016	1046	1077
Boiler Heater No. 2 Outlet		6	979	1026	1086	1106
Boiler Heater No. 2 - Boiler Heater No. 3		7	1064	1056	1075	1075
Boiler Heater No. 3 Inlet		8	1112	1105	1098	1135
Boiler Heater No. 3 Outlet		9	1114	1098	1115	1140
Boiler Heater No. 3 - Boiler Heater No. 4		10	947	1077	903	1107
Boiler Heater No. 4 Inlet		11	1106	1088	1103	1123
Boiler Heater No. 4 Outlet		12	1141	1119	1133	1137
Boiler Heater No. 4 Outlet	Immersion	14	1105	1084	1087	1116
Boiler Heater No. 4 - Superheater Heater No. 1		15	1105	1092	1102	1120
Superheater Heater No. 1 Inlet		16	1112	1187	1172	1212
Superheater Heater No. 1 Outlet		17	1141	1186	1137	1166
Superheater Heater No. 1 - Vapor Corrosion Product Separator		18	1103	1079	1088	1116
Vapor Corrosion Product Separator Inlet		20	1107	1198	1171	1188
Vapor Corrosion Product Separator Outlet		21	1106	1211	1199	1191
Vapor Corrosion Product Separator - Superheater Heater No. 2		22	1105	1113	1208	1130
Superheater Heater No. 2 Inlet		23	1280	1213	1234	1216
Superheater Heater No. 2 Outlet		24	1326	1205	1235	1261
Superheater Heater No. 2 - Superheater Heater No. 3		25	1325	1190	1219	1195
Superheater Heater No. 3 Inlet		26	1456	1336	1375	1373
Superheater Heater No. 3 Outlet		27	1423	1330	1309	1305
Throttling Valve/Orifice Inlet	Immersion	29	1092	823	768	796
Nozzle Inlet	Immersion	30	892	899	862	867
Nozzle Outlet (Specimen Holder Inlet)	Immersion	31	892	889	833	862
Specimen Holder Outlet	Immersion	32	817	808	750	787
Specimen Holder - Condenser		35	700	706	678	638
Condenser Inlet		36	636	664	637	592
Condenser Mid-Point		IC	590	660	620	576
Condenser Outlet		38	477	497	452	558
Liquid Corrosion Product Separator Inlet		39	309	499	401	527
Liquid Corrosion Product Separator Mid-Point		40	254	419	345	452
Liquid Corrosion Product Separator Outlet		41	222	362	303	397
Subcooler Inlet		42	194	310	252	339
Subcooler Mid-Point		43	161	116	184	239
Subcooler Outlet		46	131	81	141	161
Pressures and Corresponding Saturation Temperatures, psia/°F						
Boiler Outlet			302/1089	263/1064	291/1083	316/1097
Superheater Outlet			297/1086	259/1060	273/1072	314/1096
Nozzle Inlet			16.1/683	23.7/722	59.0/834	53.0/819
Condenser			13.9/669	22.5/716	30.1/749	7.0/605
Flow Rate, Pounds Per Hour			63	69	67	52

1. Run No. 1

During initial loop operation, two boiler heater failures occurred. New heaters were installed and the loop operated without incident for 693 hours. At this time, a leak developed in the bellows of the Haynes alloy No. 25 throttling valve. While testing the system for leaks, slight leakage was detected around the stem of the pneumatic-operated valve in the by-pass line around the main throttling valve. Both valves were returned to the vendor for repair.

During the interim period, heaters were installed on the liquid corrosion product separator in order to raise the temperature of this section of the system.

After receipt and installation of the two valves, the system was again leak tested. The bellows in the main throttling valve was found to be defective, and the valve was removed from the system. An orifice was fabricated from Haynes alloy No. 25 and was installed in series with the nozzle and the pneumatic-operated valve, which was moved from the by-pass line to the main line. The intention here was to use the pneumatic-operated valve to obtain fine control over the throttling process. The loop was restarted, but after several hours of operation, the pneumatic-operated valve failed and the loop was again shut down. It was then decided to operate the loop using the orifice and nozzle only, while Type 316 SS bellows were being installed in the throttling valves. It was further decided to install the valves only if satisfactory operating conditions could not be obtained with the orifice-nozzle combination. The loop was repaired and restarted incorporating these modifications.

2. Run No. 2

The loop operated satisfactorily for 66 hours, at which time a crack developed in the seam weld in the vapor corrosion product separator. The weld was re-sealed and a plate was welded over the entire length of the seam weld in the separator to give added assurance that the unit would remain leak-tight.

3. Run No. 3

The loop operated for an additional 235 hours without incident. At this time the pump began operating sporadically and the loop was shut down for repairs. The ball check valves were removed from the pump and were dismantled. A small quantity of deposit was found, primarily at the valve seats. Although the amount of deposit was extremely small, it was apparently great enough to cause sticking of the valves. The valves were cleaned, assembled, and installed and the loop was restarted. After only 19 hours of operation, a leak again developed in a weld in the vapor corrosion product separator. In order to avoid further problems with this section of the loop, a Type 316 SS shell was fabricated and installed around the entire separator.

4. Run No. 4

This run extended for a total of 4205 hours. The loop was shut down once during this period for general plant maintenance. During this run the flow varied from as low as 29 pounds per hour to as high as 130 pounds per hour, indicating that corrosion products were being formed, restricting the flow at times, with subsequent freeing of the deposits and periods of high flow. Throughout the majority of the run, the flow was stable at 50 to 60 pounds per hour. After a total of 5218 hours of operation, the test was terminated.

C. Sampling Procedure

Following shut down of the loop, the mercury was drained from the various loop sections and was collected in clean plastic containers. Because of the large quantity of mercury in the system, the mercury was removed in steps and samples were taken for analysis from each progressive draining operation.

The boiler was the first section of the loop to be drained, and was drained in three steps. One sample was reserved for analysis from each of the three 400 ml specimens removed from this section.

The next section of the loop to be drained was the condenser. One 400 ml specimen was removed from this section, and one sample was reserved for analysis.

Following the draining of these sections of the loop, the liquid corrosion product separator was removed from the system. Three 100-pound specimens of mercury were taken from this separator, followed by a fourth, nine-pound specimen. One sample was taken for analysis from each of the four specimens.

The samples of mercury reserved for analysis were exposed to air for several days in order to allow corrosion products to oxidize and separate from the mercury. Very little scum formed on the surface of any of the samples.

After removal of the mercury from the system, the vapor corrosion product separator was cut from the loop, and both separators were sectioned. The columbium alloy turnings, the iron wool, and the deposits found in the separators were submitted for chemical analysis.

The loop was then cut into sections and each section was cut longitudinally for evaluation. Figure 10 is a schematic of the loop, identifying the various sections removed for analyses.

Upon sectioning the loop, deposition was revealed in various locations. All deposits in the system were collected and submitted for analysis. Specimens were also removed from the loop and were submitted for metallurgical evaluation.

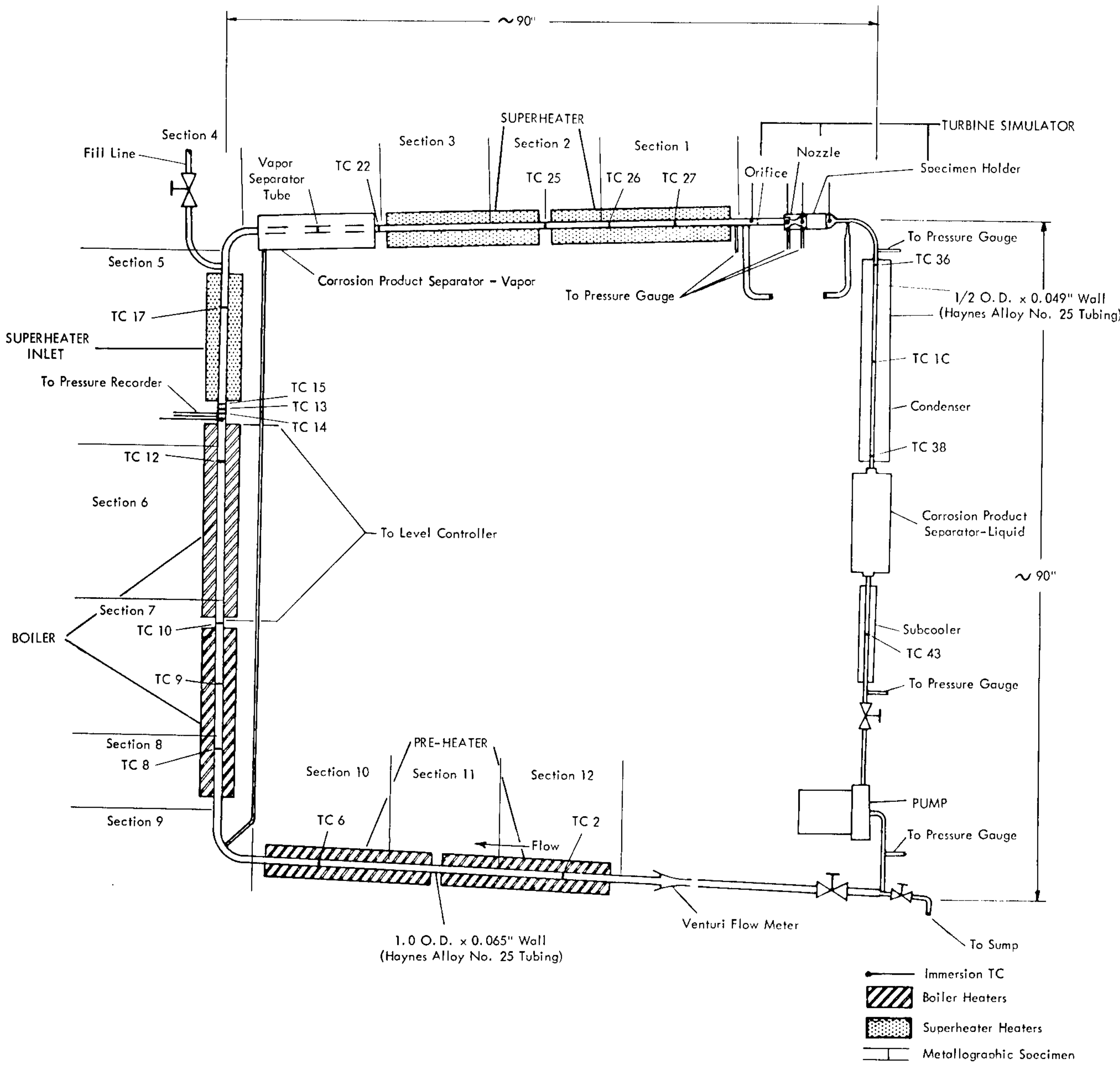


FIGURE 10. SCHEMATIC OF LOOP SHOWING SECTIONING LOCATIONS AND LOCATION OF METALLOGRAPHIC SPECIMENS

RESULTS AND DISCUSSION

A. Visual Observations

As shown in Figures 11 and 12, the Venturi was found to contain an amalgam which was located on the outlet side in the vicinity of the orifice. The pre-heater (Sections 10, 11, and 12) also contained an amalgam, as shown in Figure 13. Photographs of the boiler are shown in Figure 14. As seen here, the boiler inlet contained a small quantity of a dark deposit while the mid-section and outlet again contained an amalgam. The amalgam extended into the superheater inlet, as shown in Figure 15. From this point on, as the mercury was dried and superheated, the quantity of deposit decreased until the superheater outlet was reached, where an amalgam was again present. The vapor corrosion product separator outlet tube and the superheater mid-section were essentially free of deposit, Figure 15.

A hard deposit was found in the orifice and is shown in Figure 16. The deposit contained essentially no mercury and was blocking approximately 99.2 percent of the flow passage. The presence of this deposit accounts for the decrease in flow rate in the system during the final hours of operation. However, it is believed that the majority of this deposit was formed during the shut-down operation, since the change in flow rate did not reflect essentially complete blockage.

The nozzle was also observed to contain a deposit, as shown in Figure 17. Although there was a small amount of deposition on the inlet side, the majority of the deposit was found on the outlet.

The PH15-7Mo and Nivco 10 erosion specimens located at the nozzle outlet suffered no gross attack or deposition. These specimens are shown in Figure 18.

The condenser and subcooler tubing is shown in Figure 19. As seen here, these sections of the loop were quite clean.

B. Chemical and Metallurgical Evaluations

1. Corrosion

Locations of the various loop sections examined metallographically are shown in Figure 10, which is reproduced on page 31 for convenience. Metallographic results are summarized in Tables 2 through 4 and representative photomicrographs are presented in Figures 20 through 28. Loop section and thermocouple locations are indicated for each photomicrograph.

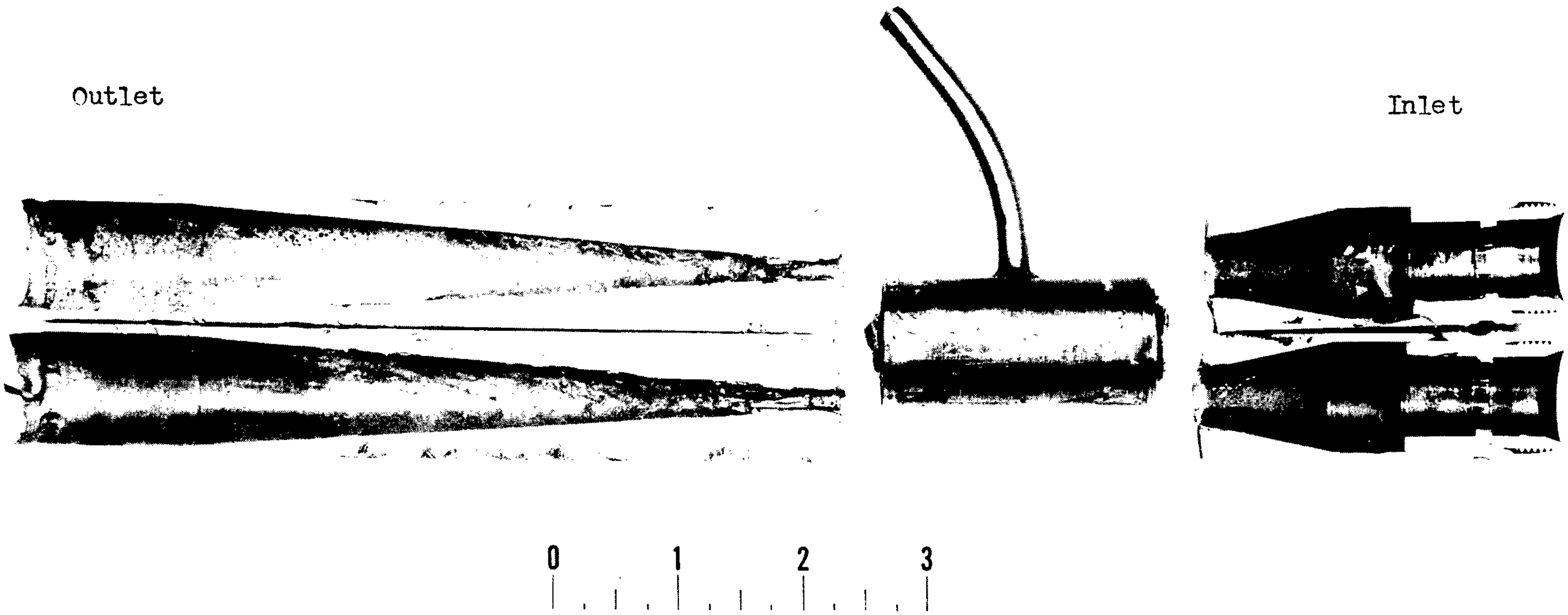
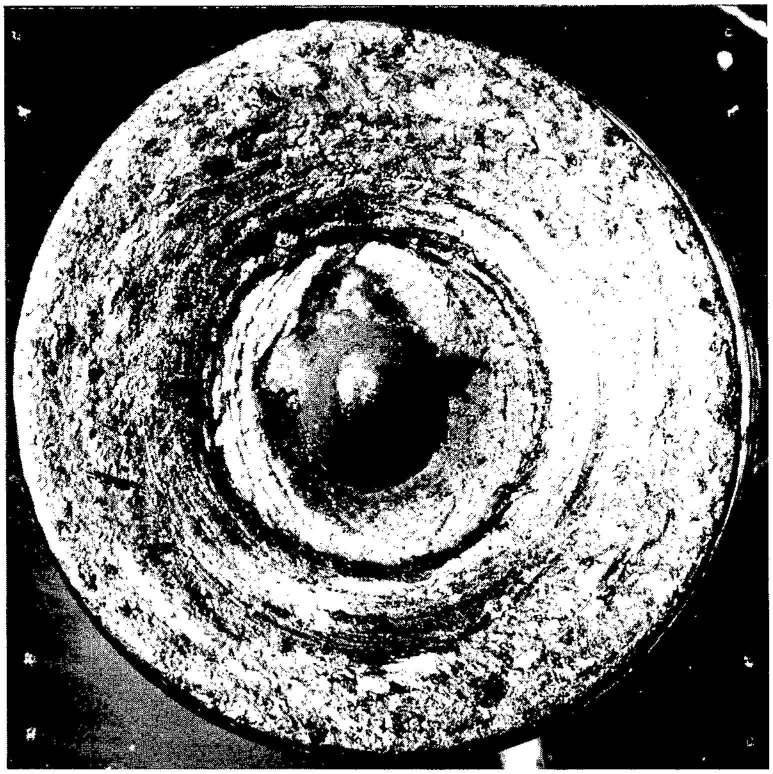
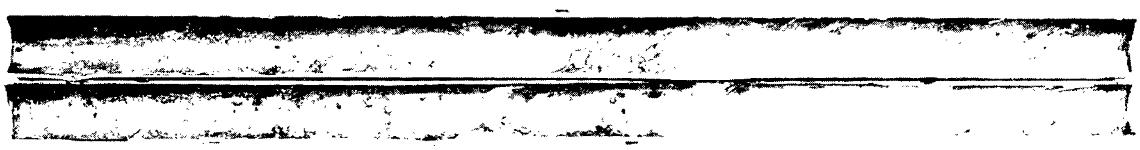


Figure 11. Deposition in Venturi.



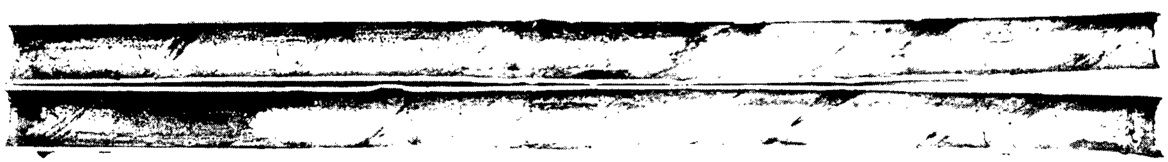
4X

Figure 12. Outlet Side of Venturi Orifice Showing Amalgam Deposit.



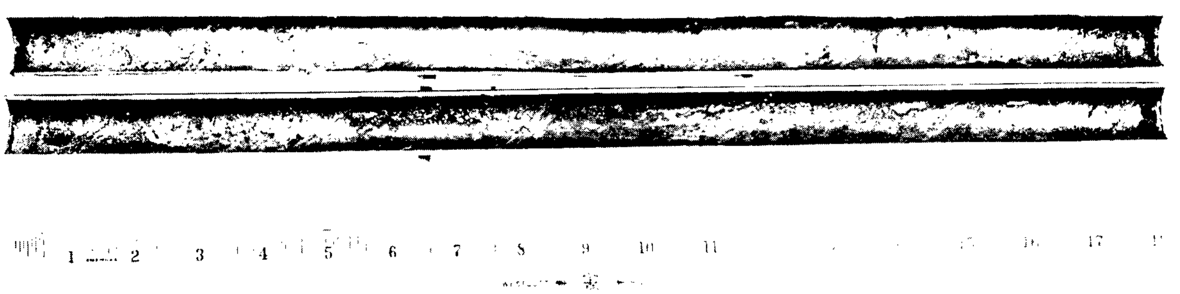
Flow

Section 12 - Pre-Heater Inlet



Flow

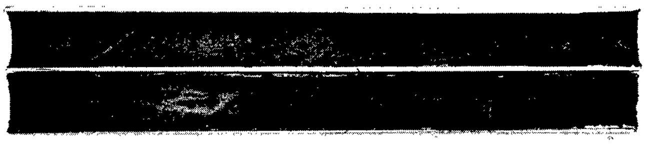
Section 11 - Pre-Heater Mid-Section



Flow

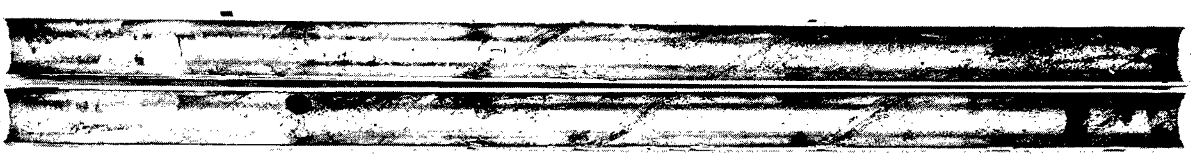
Section 10 - Pre-Heater Outlet

Figure 13. Pre-Heater Section of Loop.



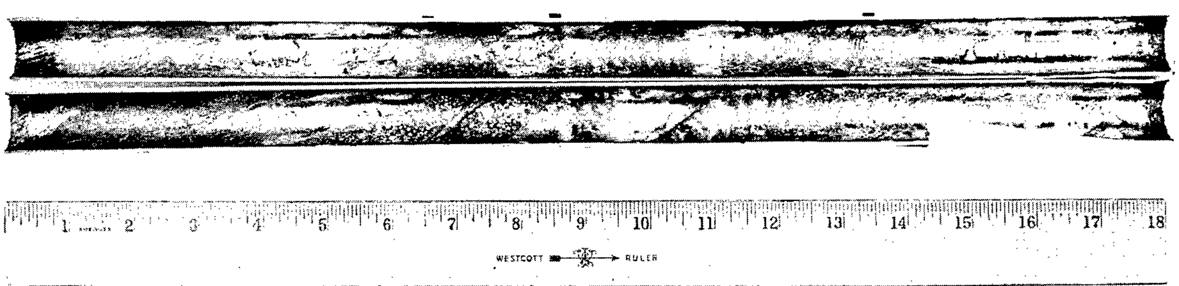
Flow

Section 8 - Boiler Inlet



Flow

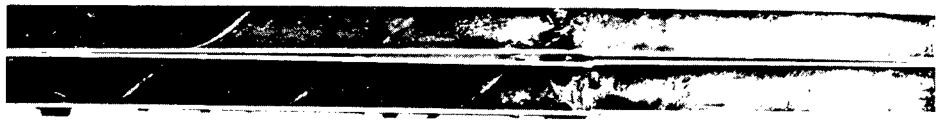
Section 7 - Boiler Mid-Section



Flow

Section 6 - Boiler Outlet

Figure 14. Boiler Section of Loop.

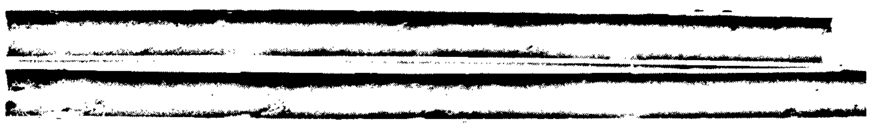


Flow

Section 5 - Superheater Inlet

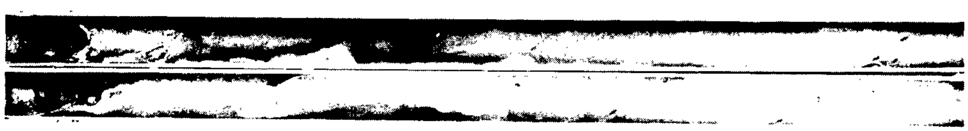
Flow

Section 3 - Vapor Corrosion Product Separator Outlet Tube



Flow

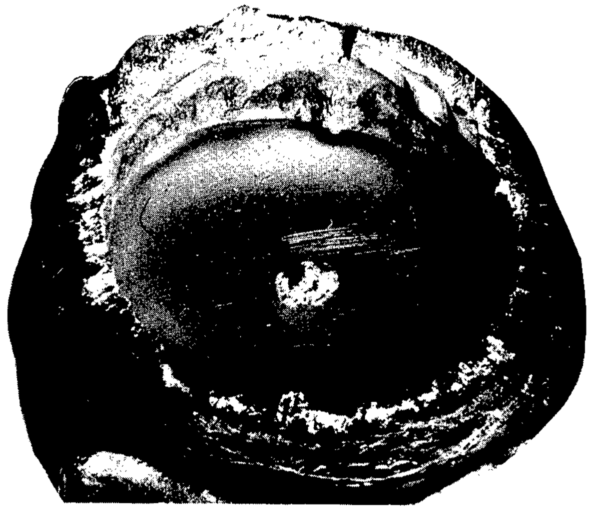
Section 2 - Superheater Mid-Section



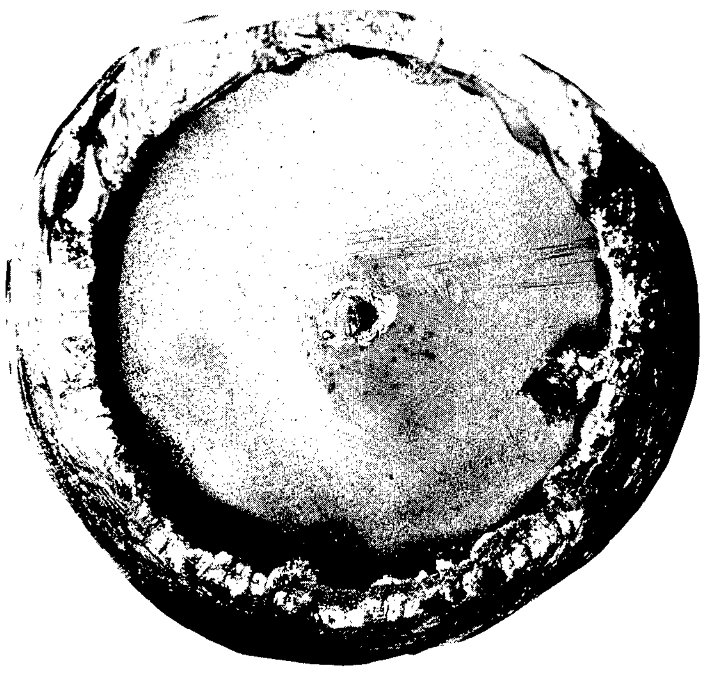
Flow

Section 1 - Superheater Outlet

Figure 15. Superheater Section of Loop.

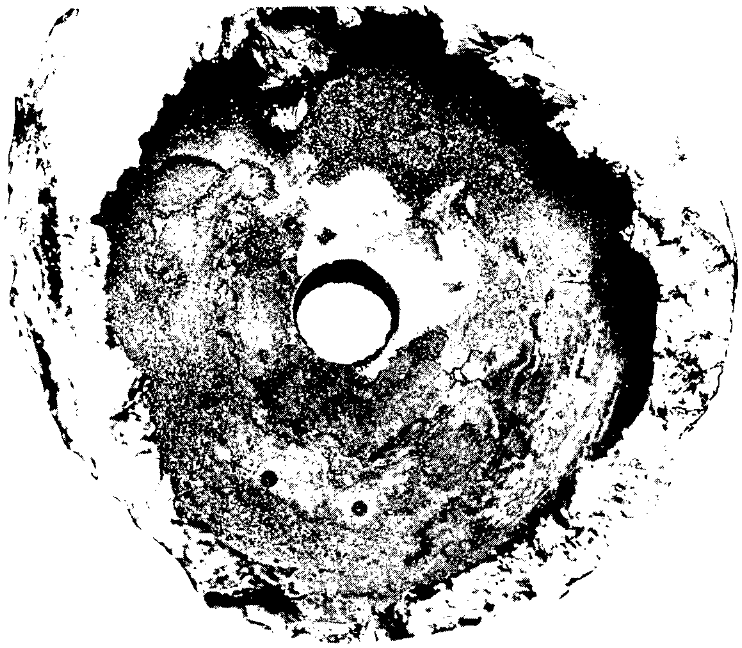


a) Inlet Approx. 2X



b) Outlet Approx. 2.5X

Figure 16. Deposit Found in Loop Orifice.

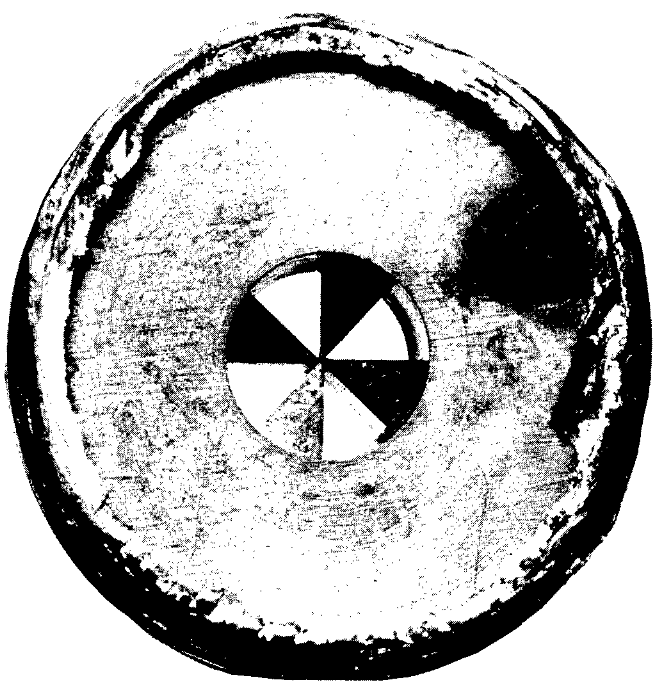


a) Inlet Approx. 3X

b) Outlet Approx. 3X

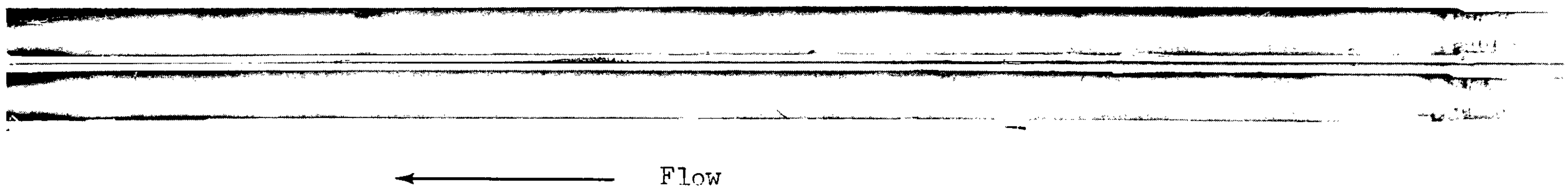
Figure 17. Deposit Found in Loop Nozzle.

a) Inlet Approx. 3X

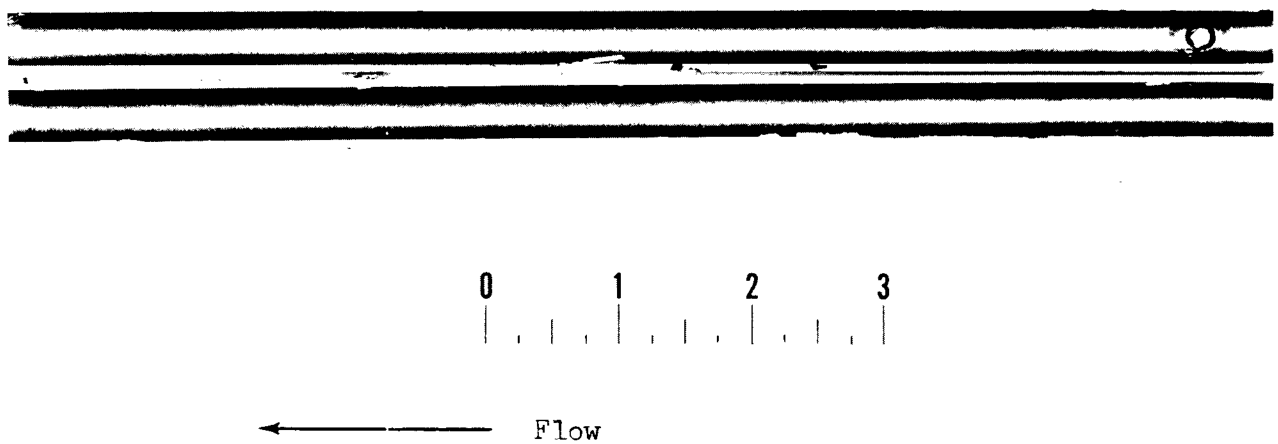


b) Outlet Approx. 3X

Figure 18. Erosion Specimen Holder.



a) Condenser Section



b) Subcooler Section

Figure 19. Condenser and Subcooler Sections of Loop.

TABLE 2

RESULTS OF METALLOGRAPHIC EVALUATION OF LOOP

Location	Thermocouple Number	Temp. °F	Maximum Corrosion Mils	Maximum Corrosion Type*	Remarks
Pre-Heater Inlet	2	838	1.85	C	Liquid
Pre-Heater Outlet	6	1106	10.0	L	
Boiler Inlet	8	1135	4.7	L	
Boiler Mid-Section	9	1140	3.85	L&I	
Boiler Mid-Section	10	1107	2.5	L	
Boiler Outlet	12	1137	1.1	L	Liquid and vapor
Superheater Inlet	13	1118	7.0	L	
Superheater Inlet	14	1116	-	-	Weld crack
Superheater Inlet	15	1120	1.35	L	
Superheater Inlet	17	1166	2.75	L	
Vapor Separator Tube	-	~1130	1.9	I	Vapor and liquid No attack on weld
Vapor Separator Outlet	22	1130	0.2	C	
Superheater Mid-Section	25	1195	1.1	C	
Superheater Outlet	26	1373	1.1	C	
Superheater Outlet	27	1305	0	-	7 Mil deposit
Orifice**		796-867	7.26	L&I	Deposit on outlet side
Nozzle		867-862	5.0	I	Deposit on converging section
Condenser Inlet	36	592	0.55	C&L	
Condenser Mid-Section	IC	576	0.3	C	
Condenser Outlet	38	558	0.95	C&L	
Liquid Separator	-	-	0	-	
Subcooler	43	239	0.55	C	Liquid

*L - layer
C - crevice
I - intergranular

**Nivco 10

TABLE 3

RESULTS OF METALLOGRAPHIC EVALUATION OF SWIRL WIRE

Location	Section	Maximum Corrosion Mils	Maximum Corrosion Type*	Remarks
Pre-Heater Inlet	12	0.55	C	Liquid
Pre-Heater Mid-Section	11	3.6	L	
Pre-Heater Outlet	10	2.75	L	
Boiler Inlet	8	0.55	L	Deposition
Boiler Mid-Section	7	2.4	L&I	
Boiler Outlet	6	0.8	C	Cracking. Liquid and vapor
Superheater Inlet	5	0.8	C	Cracking
Superheater Inlet	4	1.35	L	Cracking
Vapor Corrosion Product Separator Outlet Tube	3	0.5	I	Cracking
Superheater Mid-Section	2	0.8	I	Cracking
Superheater Outlet	1	0.3	I	

*L - layer
C - crevice
I - intergranular

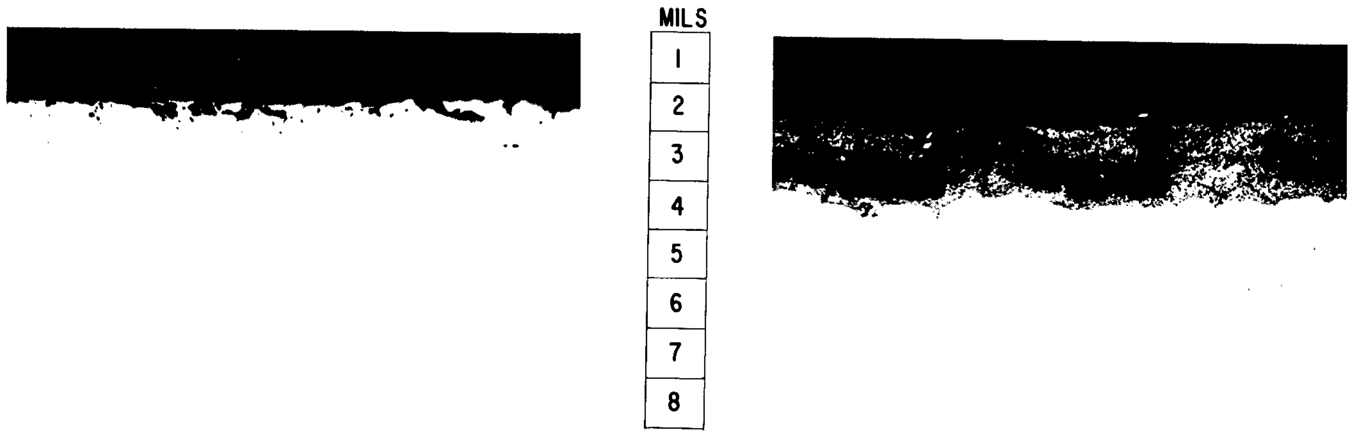
TABLE 4

EROSION SPECIMEN EVALUATION FROM VAPOR NOZZLE OUTLET

No.	Material	Corrosion Mils	Corrosion Type*	Erosion Mils	Remarks
1	Nivco 10	2.5	I	0	Crack-like penetrations
2	PH15-7Mo(TH1050)	0		2.5	
3	Nivco 10	2.2	I	1.85	Crack-like penetration, deposition
4	PH15-7Mo (RH 950)	0.5	C	1.85	

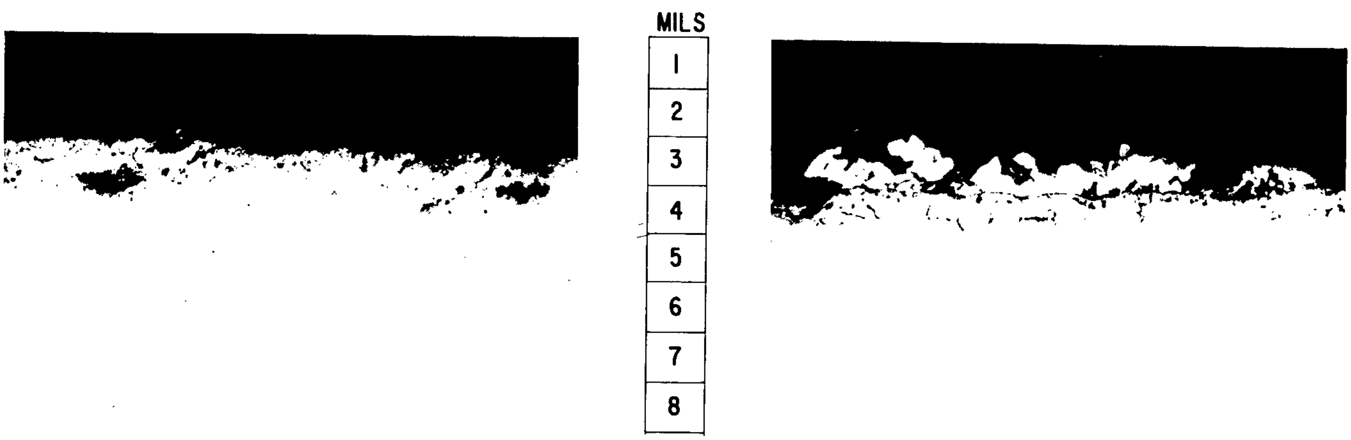
*I - intergranular

C - crevice



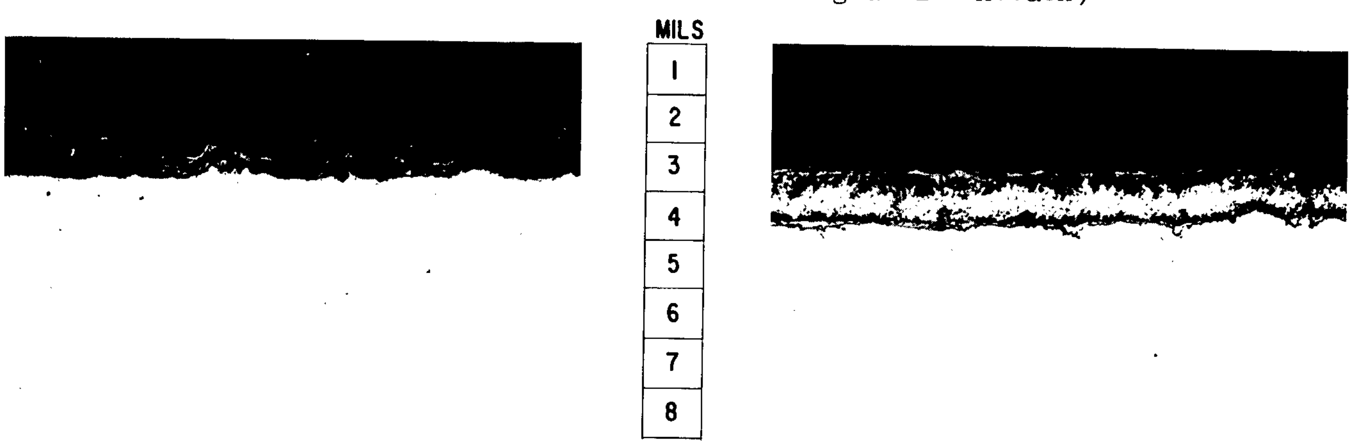
a) Section 12, TC 2 Pre-Heater Inlet (Crevice Attack)

b) Section 10, TC 6 Pre-Heater Outlet (Layer Attack)



c) Section 8, TC 8 Boiler Inlet (Layer Attack)

d) Section 7, TC 9 Boiler Mid-Section (Deposition and Layer and Inter-granular Attack)

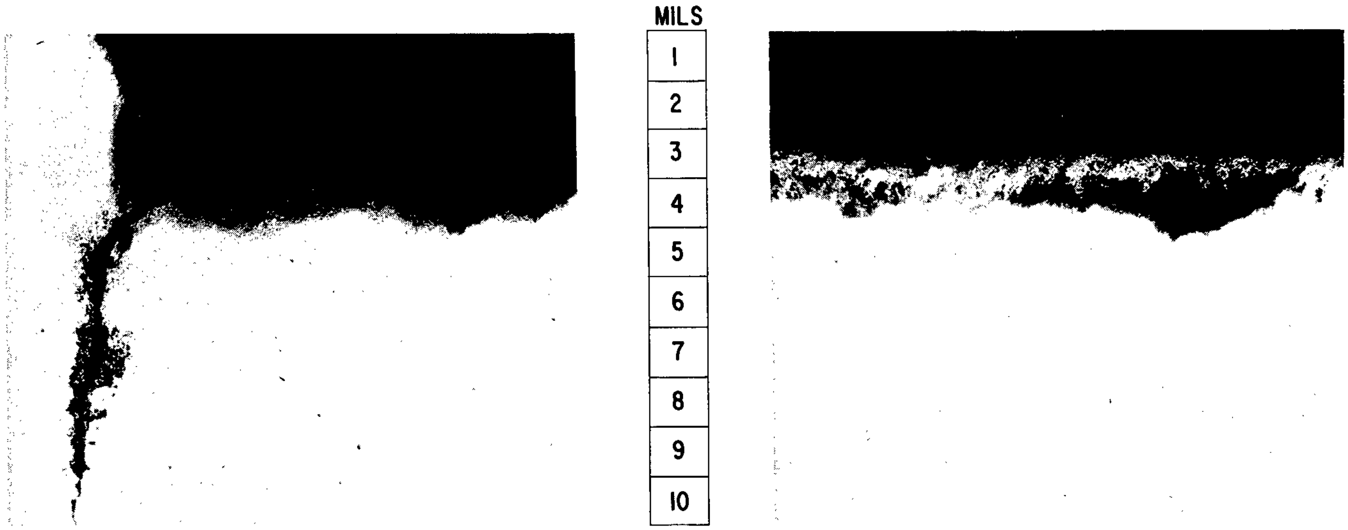


e) Section 7, TC 10 Boiler Mid-Section (Layer Attack)

f) Section 6, TC 12 Boiler Outlet (Layer Attack)

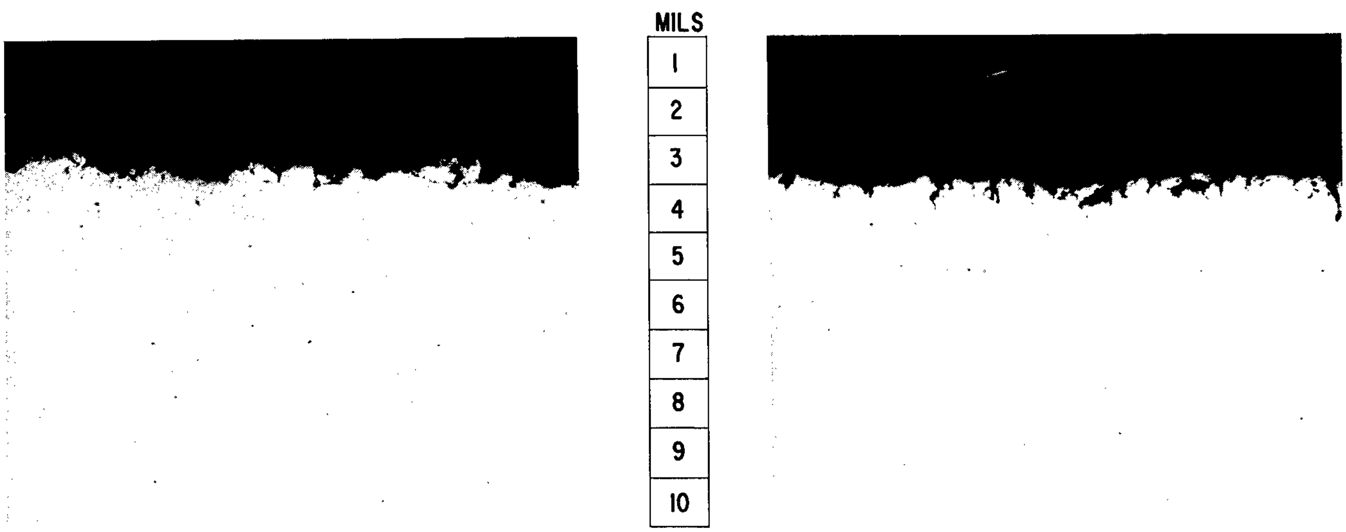
Figure 20. Typical Cross-Sections of Loop Pre-Heater and Boiler.

Magnification: 250X, Unetched.



a) Section 5, TC 14
Weld; Superheater Inlet
(Layer Attack)

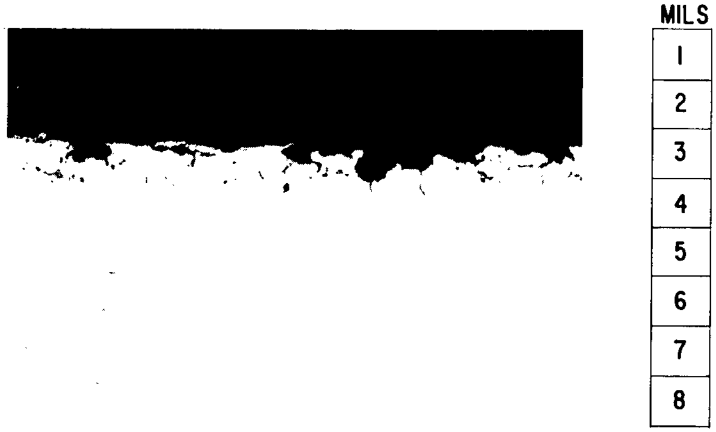
b) Section 5, TC 14
Superheater Inlet
(Layer Attack)



c) Section 5, TC 17
Superheater Inlet
(Crevice Attack)

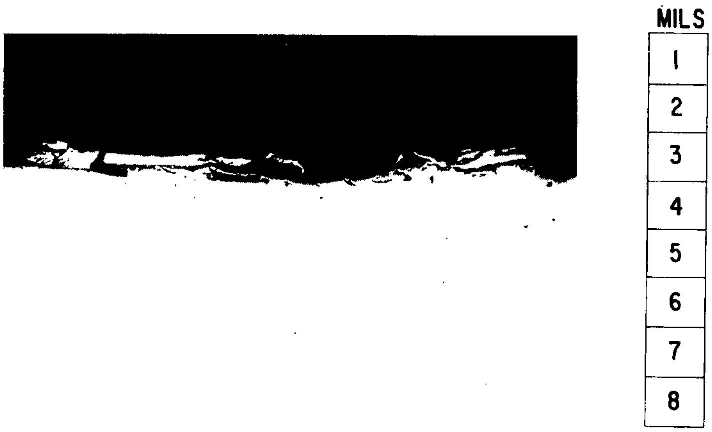
d) Vapor Corrosion Product
Separator Filter Tube
(Crevice and Intergranular Attack)

Figure 21. Typical Cross-Sections of Loop Superheater Inlet and Vapor Corrosion Product Separator Filter Tube. Magnification 250X, Unetched.



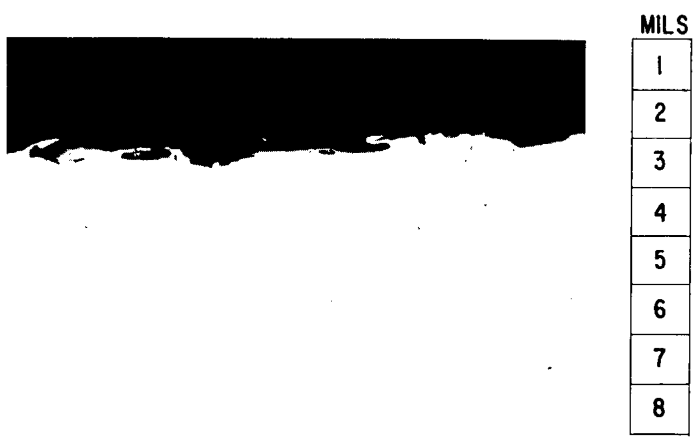
a) Section 2, TC 25
Superheater Mid-Section
(Crevice Attack)

b) Section 1, TC 27
Superheater Outlet
(Deposition)



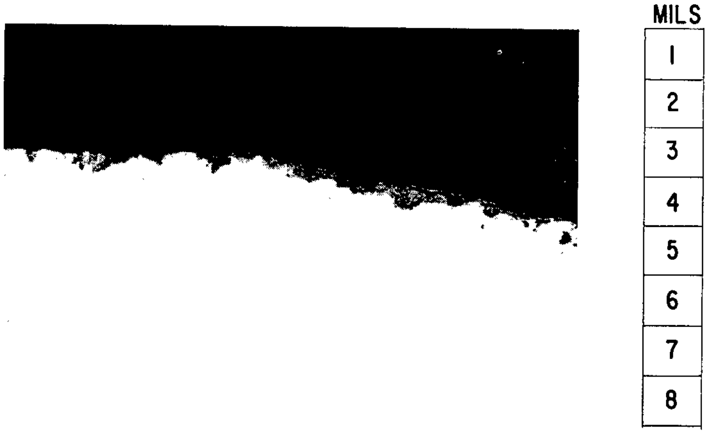
c) TCIC
Condenser Mid-Section
(Crevice Attack)

d) TC 38
Condenser Outlet
(Crevice and Layer Attack)



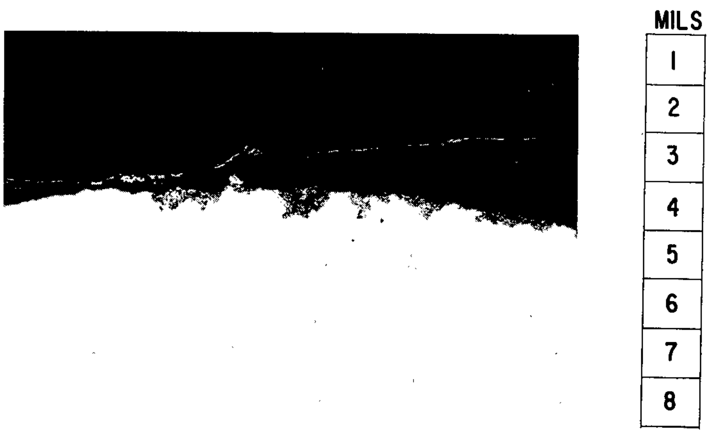
e) TC 43
Subcooler Mid-Section
(Crevice Attack)

Figure 22. Typical Cross-Sections of Loop Superheater, Condenser and Subcooler. Magnification: 250X, Unetched.



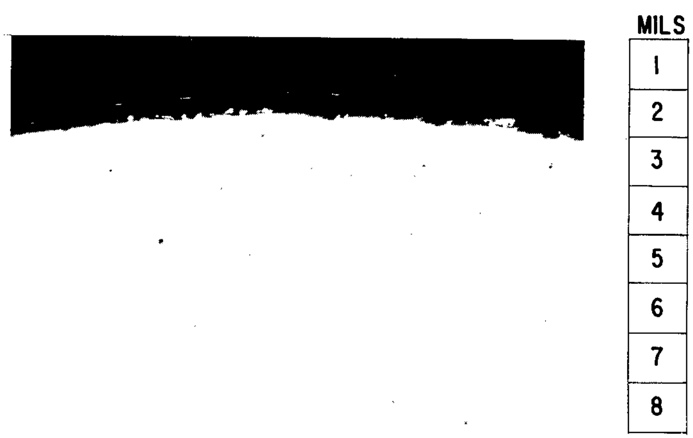
a) Section 12
Pre-Heater Inlet
(Crevice and Layer Attack)

b) Section 11
Pre-Heater Mid-Section
(Layer Attack)



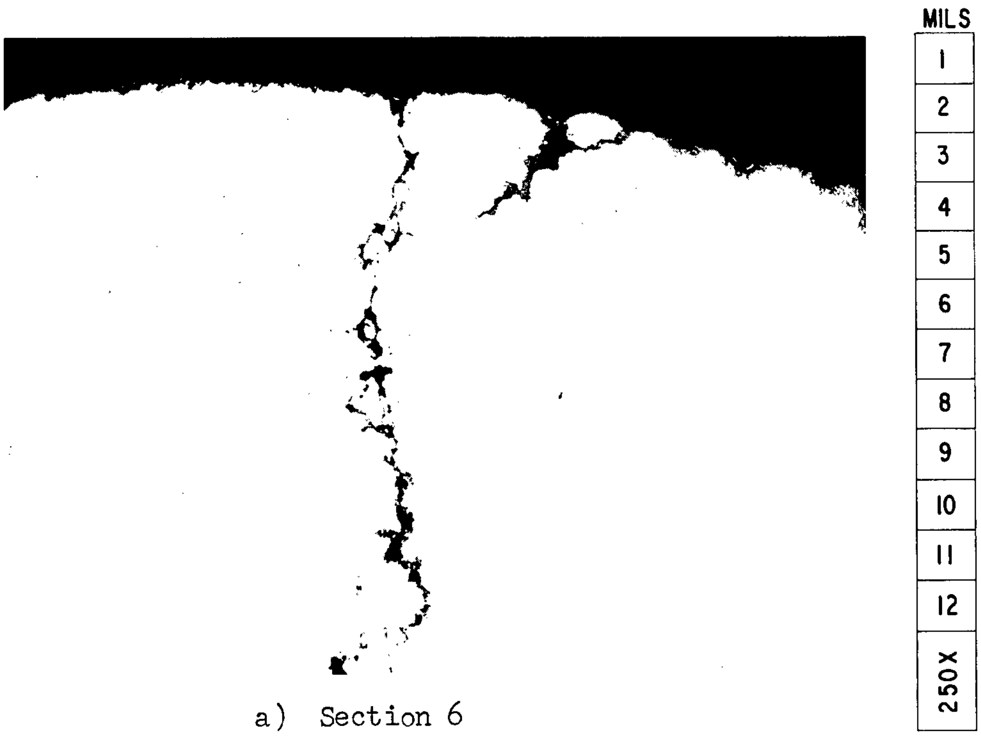
c) Section 8
Boiler Inlet
(Layer Attack)

d) Section 7
Boiler Mid-Section
(Layer and Intergranular Attack)

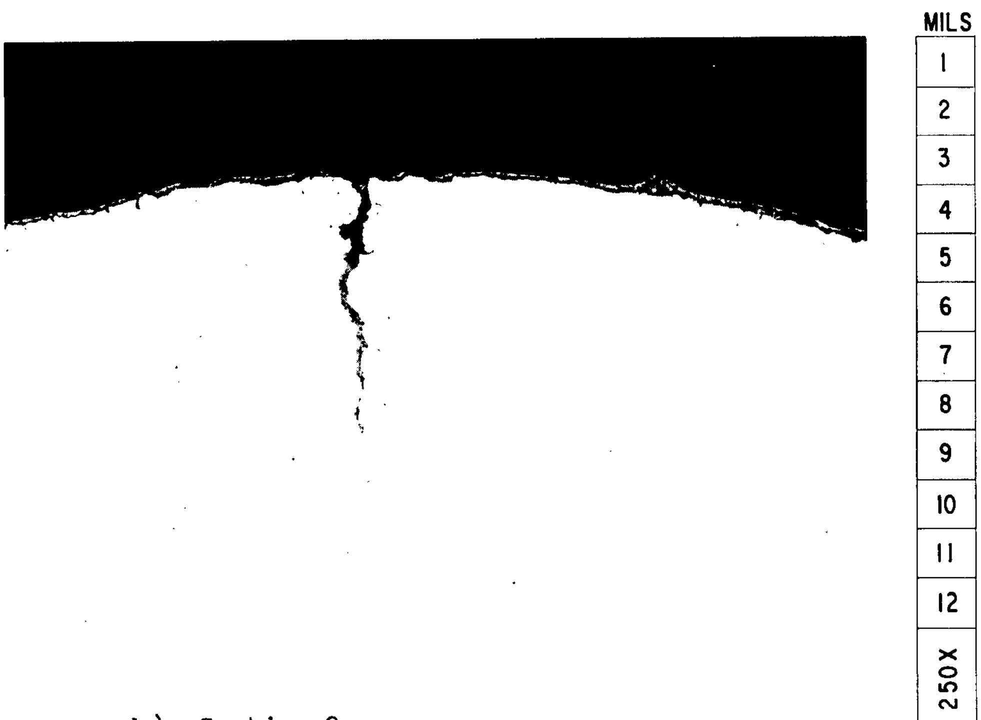


e) Section 1
Superheater Outlet
(Deposition and Intergranular Attack)

Figure 23. Typical Transverse Microstructures of Swirl Wire.
Magnification: 250X, Unetched.



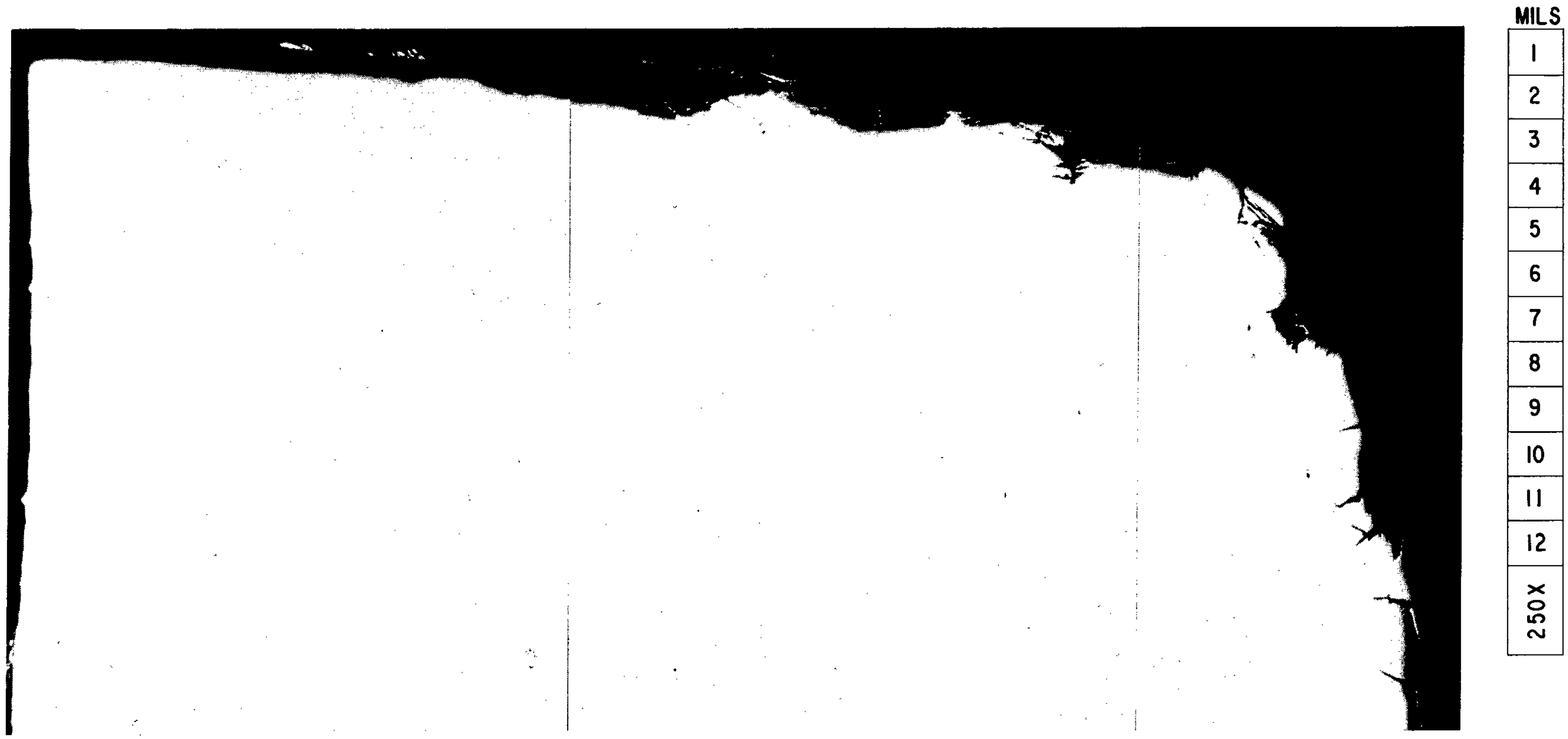
a) Section 6
Boiler Outlet
(Cracking and Layer
and Crevice Attack)



b) Section 2
Superheater Mid-Section
(Cracking and Intergranular Attack)

Figure 24. Typical Cross-Sections of Swirl Wire.
Magnification: 250X, Unetched.

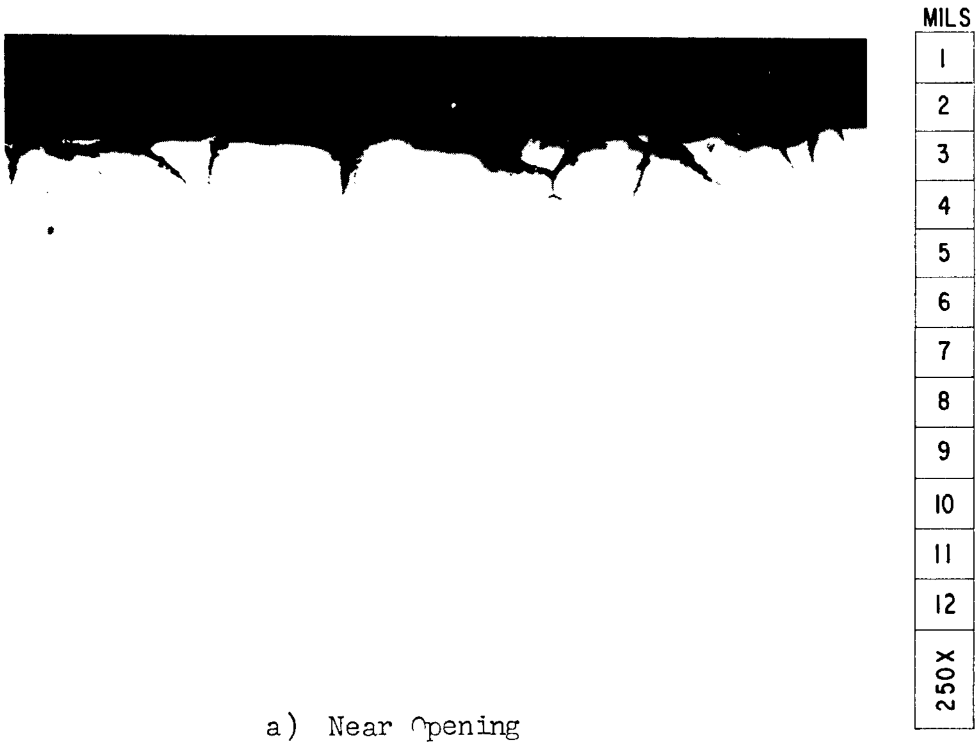
Orifice Opening



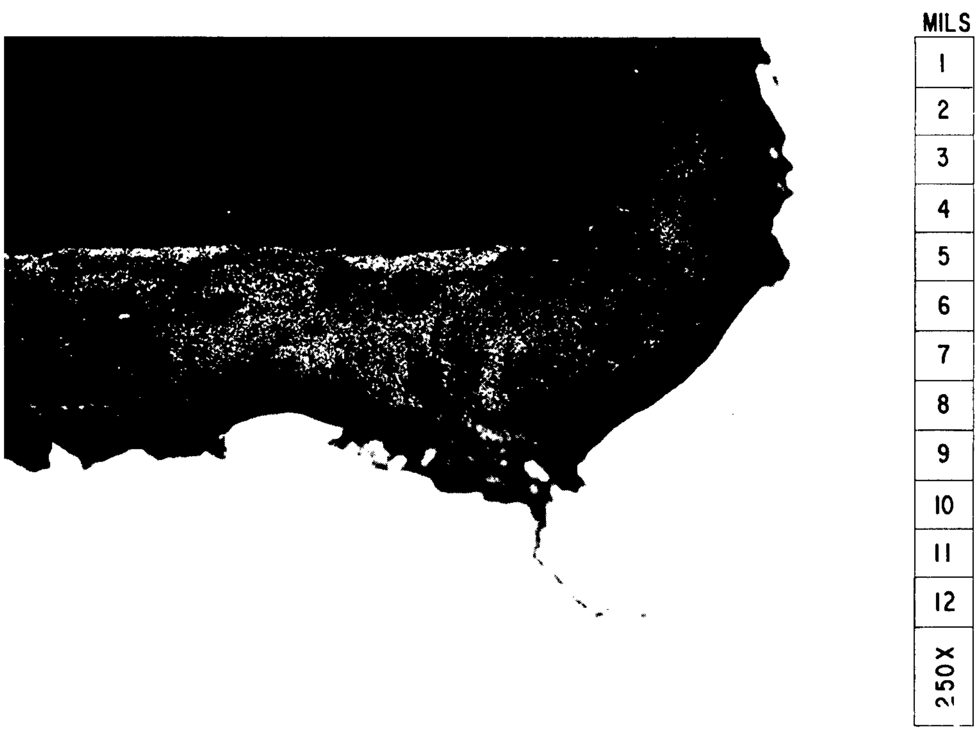
Outlet Side

Inlet Side

Figure 25. Cross-Section of Loop Orifice (Haynes Alloy No. 25).
Magnification: 250X, Unetched.
(Layer and Intergranular Attack)

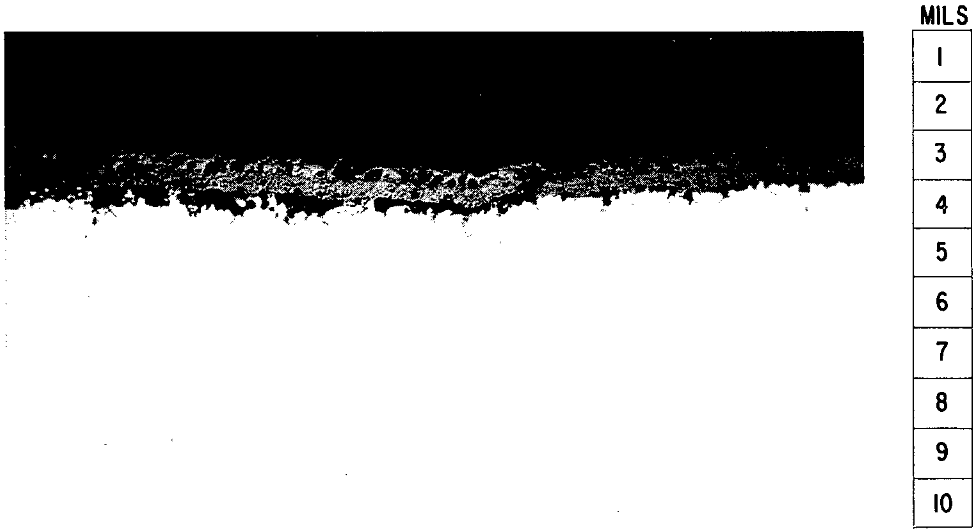


a) Near Opening

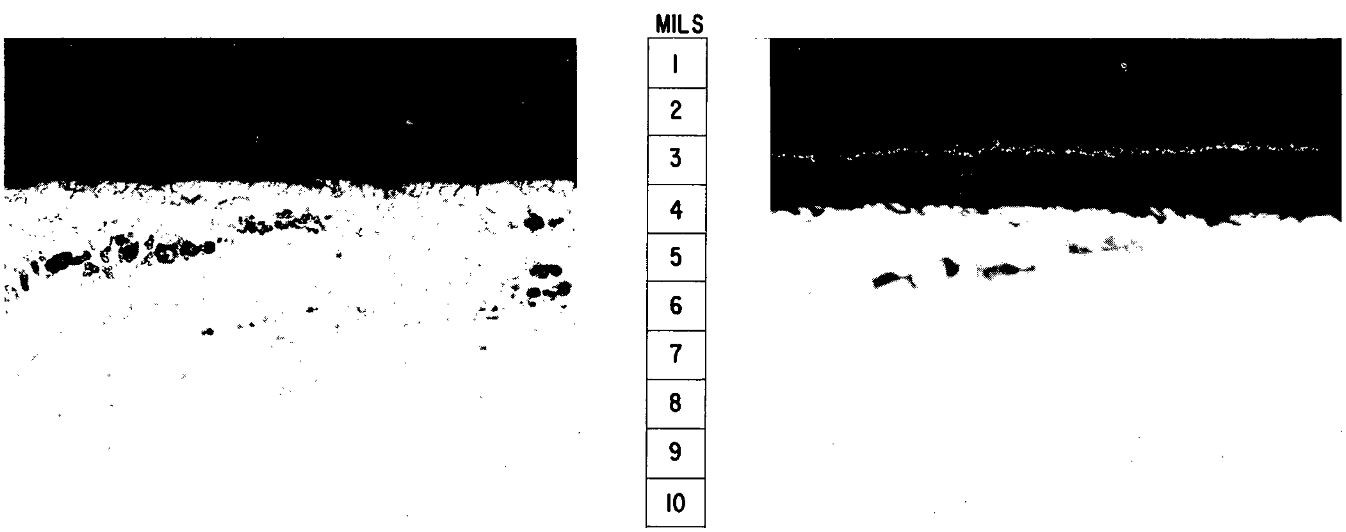


b) At Tube-Orifice Plate Junction

Figure 26. Cross-Section of Orifice on Inlet Side Showing Intergranular Attack Near Orifice Opening and a Leached Layer and Cracking at the Orifice Plate-Tube Junction. Magnification: 250X, Unetched.



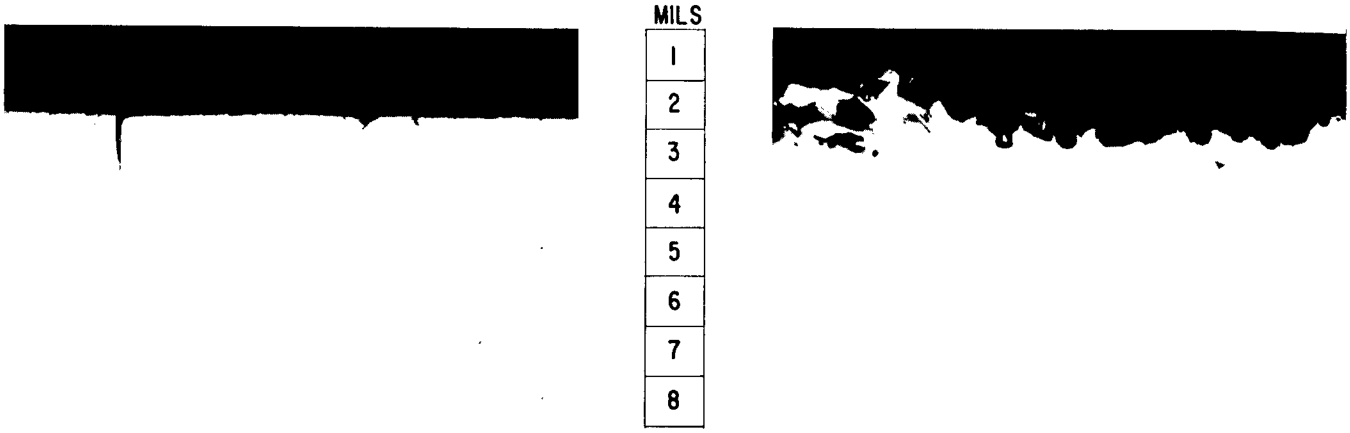
a) Inlet Side Oxidation



b) Throat Area
(Intergranular Attack

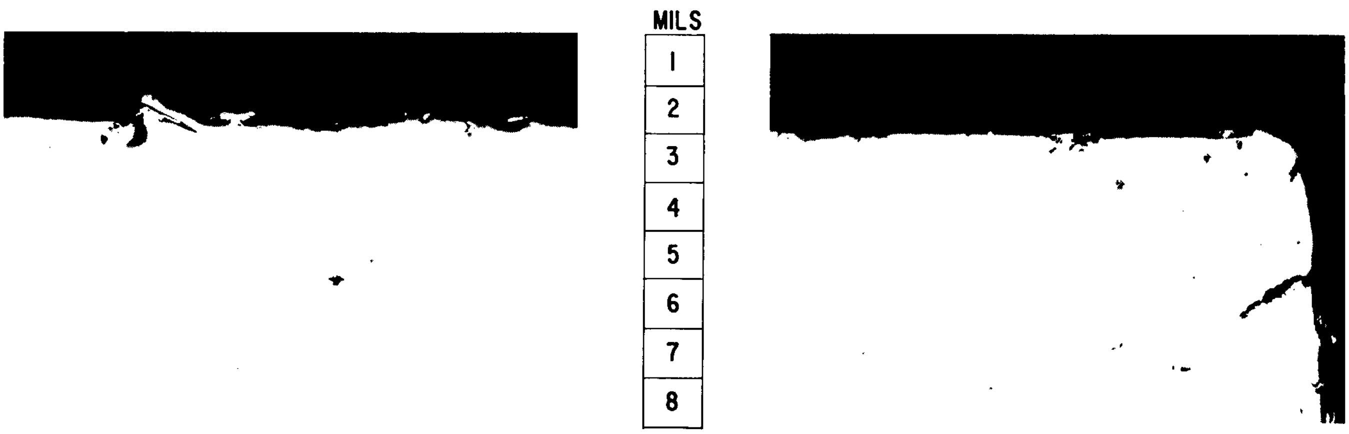
c) Throat Area
(Deposition and
Intergranular Attack)

Figure 27. Cross-Sections of Nivco 10 Nozzle Inlet Side and Throat Showing Oxidation, Intergranular Penetrations, and Deposits. Magnification: 250X, Unetched.



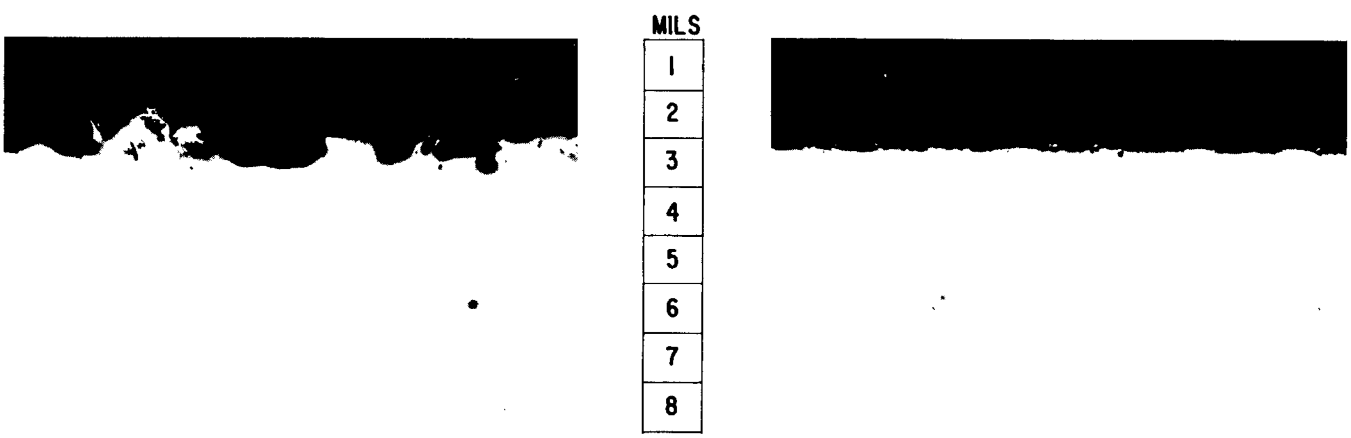
a) No. 1 Nivco 10
(Intergranular Attack)

b) No. 2 PH15-7Mo (TH 1050)
(Erosion)



c) No. 3 Nivco 10
(Erosion and
Intergranular Attack)

d) No. 3 Nivco 10
(Deposition and
Intergranular Attack)



e) No. 4 PH15-7Mo (RH 950)
(Erosion)

f) No. 4 PH15-7Mo (RH 950)
(Crevice Attack)

Figure 28. Cross-Sections of Erosion Specimens. Magnification: 250X, Unetched.

Corrosion in the horizontal pre-heater inlet section of the loop was manifested as crevice attack up to 1.85 mils deep. A typical photomicrograph is shown in Figure 20a. The average wall temperature at this section was measured at 838°F. Swirl wire attack was less severe, 0.55 mils, Figure 23a and Table 3. The attack was similar to the container wall attack (i.e., crevice type).

The pre-heater outlet section suffered the most serious attack observed in the loop (see Figure 20b), and was as great as 10 mils in the 1106°F region, Table 2. Attack was of the leached zone type. Swirl wire attack was again considerably less severe (2.8 mils) than the corresponding wall attack and is represented by Figure 23b.

Attack in the vertical section of the boiler and superheater inlet decreased from 4.7 to 2.5 mils in the direction of increased vapor generation, Figures 20c-f and 21a-c. However, some areas ranged as high as seven mils in penetration, Table 2. The wall temperature varied between 1107 and 1140°F in these sections, and attack was of the leached layer type. Swirl wire penetration varied between 0.55 and 2.4 mils. Attack was both crevice and leached zone type, Figures 23c, 23d, and 24a. In the vapor portion of the superheater inlet section, the attack depth varied generally between 1.35 and 2.75 mils, Table 2.

The vapor corrosion product separator was attacked to a depth of 1.9 mils in the 1130°F temperature zone. Corrosion was manifested as both intergranular and leached layer attack (see Figure 21d). Attack diminished to 1.1 mils penetration in the superheater mid-section as vapor quality improved, Figure 22a. Corrosion was of the crevice type. In the last quarter of the superheater (outlet), a substantial deposit was observed, Figure 22b. The maximum thickness of this deposit was seven mils. Swirl wire attack in the vapor portion of the loop generally was of the intergranular type and varied between 0.3 and 1.35 mils, Figures 23e and 24b and Table 3. Only the Haynes alloy No. 25 swirl wire in the vapor region contained cracks as contrasted to the lack of cracking in the liquid-contacted sections, Figure 24. The swirl wire was cold-worked as a result of swirl forming.

Haynes alloy No. 25 fusion welds appeared to be unattacked in all cases except in the vapor section (No. 5) of the boiler. Corrosion was present at the weld and in the crack, Figure 21a. Corrosion was manifested as a depleted layer.

Orifice corrosion was greatest on the inlet side near the wall and measured 7.3 mils in depth, Figure 25. Cracking was also observed at the orifice plate-tube wall junction, Figure 26b. Corrosion up to 1.1 mils in depth was also present on the orifice opening, Figure 26a. The corrosion was manifested as both layer and intergranular attack. The outlet side of the orifice contained deposits which adhered to the orifice wall. The attack was undoubtedly a result of mercury vapor condensation on the orifice plate, since immersion thermocouples recorded 796 and 867°F as the respective inlet and outlet temperatures. The severity of the attack could also be a result of erosive action by the condensing mercury vapor, which is not readily separable from corrosive attack.

The nozzle, made from Nivco 10, suffered up to five mils of penetration on both the inlet and outlet sides. (Immersion thermocouples recorded 867 and 862°F as the inlet and outlet temperatures, respectively, of the mercury vapor, indicating that condensation was also occurring in this area.) The corrosion was manifested as intergranular penetration, Figure 26b. Some deposition was also apparent on the converging nozzle surfaces, Figure 27b. Heavy atmospheric oxidation was also evident on the nozzle inlet side, Figure 27a. The layer in the throat section could also be an oxide coating, Figure 27c. Oxidation undoubtedly occurred during the several shut down periods resulting from the faulty throttling valves. Although the loop was protected with an argon cover during these periods, complete protection could not be given to this region of the system until the loop had cooled to room temperature.

Condenser corrosion varied from 0.4 to 0.95 mils, the greatest penetrations being observed in the condenser outlet, Figure 22d. Corrosion was evident as crevice attack for all condenser sections evaluated.

Subcooler penetrations averaged 0.55 mils in depth and they were of the crevice type, Figure 22e.

An electron beam microanalysis was performed by Advanced Metals Research Corporation on three of the specimens taken from the loop. The specimens examined were the pre-heater outlet (see Figure 20b), the condenser outlet (see Figure 22d), and the nozzle throat (see Figure 27b). The results of the electron beam microanalysis confirmed the metallographic results and are presented in the Appendix. The analysis of the pre-heater and condenser specimens included the elements columbium, titanium, and zirconium because of the presence of these elements in the columbium alloy getter used in the corrosion product separators. No mass transfer of these elements was revealed by the microanalysis.

The erosion specimens were mounted on one of the flat surfaces so that the knife edge and one edge exposed to the 862°F mercury vapor could be examined for erosion and corrosion effects. The corrosion and erosion damage was evaluated separately, although, in some cases the two degradation processes tended to compound one another and thus could not be distinguished from each other.

Corrosion was observed on three specimens, namely Nos. 1, 3, and 4 (Table 4). Corrosion on the PH15-7Mo in condition RH 950 (sample No. 4) was manifested as a leached layer less than one mil deep, Figure 28f. Corrosion on the Nivco 10 was manifested as crevice attack on specimen No. 3 and as intergranular penetrations up to 2.5 mils in depth on specimen Nos. 1 and 3, Figures 28a, 28c, and 28d. Deposits of corrosion products were also evident on Nivco 10 specimen No. 3, Figure 28d.

What appeared to be erosion damage occurred on three specimens, namely Nos. 2 (PH15-7Mo), 3 (Nivco 10), and 4 (PH15-7Mo).

Erosion damage was heaviest on PH15-7Mo in condition TH 1050 (specimen No. 2). An estimated 2.5 mils was eroded as shown in Figure 28b.

Corrosion was also evident in the eroded area. Erosion damage on PH15-7Mo in condition RH 950 (specimen No. 4) was less severe, 1.85 mils, Figure 28e. The increased resistance to erosion of specimen No. 4 over specimen No. 2 was probably the result of the greater hardness of specimen No. 4 (about 3 Rockwell C hardness units). Nivco 10 specimen No. 1 showed no signs of erosion. However, specimen No. 3 showed erosion up to 1.85 mils, Figure 28c.

Unfortunately, when the erosion specimen holder was cut open, the specimens were also cut and a weight change could not be determined. Hardness readings were taken, however, before and after the test and are given in Table 5. These readings indicate that the PH15-7Mo changed very little, while the hardness of the Nivco 10 increased considerably. As a point of interest, the hardness of the PH15-7Mo specimen in the RH 950 condition was used to approximate the average temperature of this specimen during the test, using a master aging curve for this material, Figure 29 (3). Using this method, an average temperature of 875°F was calculated. This compares quite favorably with the 862°F temperature indicated by an immersion thermocouple located at the specimen holder inlet.

Corrosion for the Haynes alloy No. 25 forced circulation corrosion loop was compared with maximum penetration data obtained from thermal convection two-phase loops (4). Although the mercury mass flow rate was on the order of 10 to 40 pounds per hour greater in this forced circulation loop than in the natural circulation loops, the data compare favorably, as shown in Figure 30. The majority of the points fall below the maximum penetration observed for two-phase thermal convection mercury loops. The points which lie above this curve are the two isolated penetrations observed in the pre-heater outlet (10 mils at 1106°F) and the superheater inlet (7 mils at 1118°F).

TABLE 5

HARDNESS READINGS ON LOOP EROSION SPECIMENS

Specimen	Average Hardness, R_c Before Test	After Test
PH15-7Mo (RH 950)	49	48
PH15-7Mo (TH 1050)	46	48
Nivco 10	31	36
Nivco 10	30	35

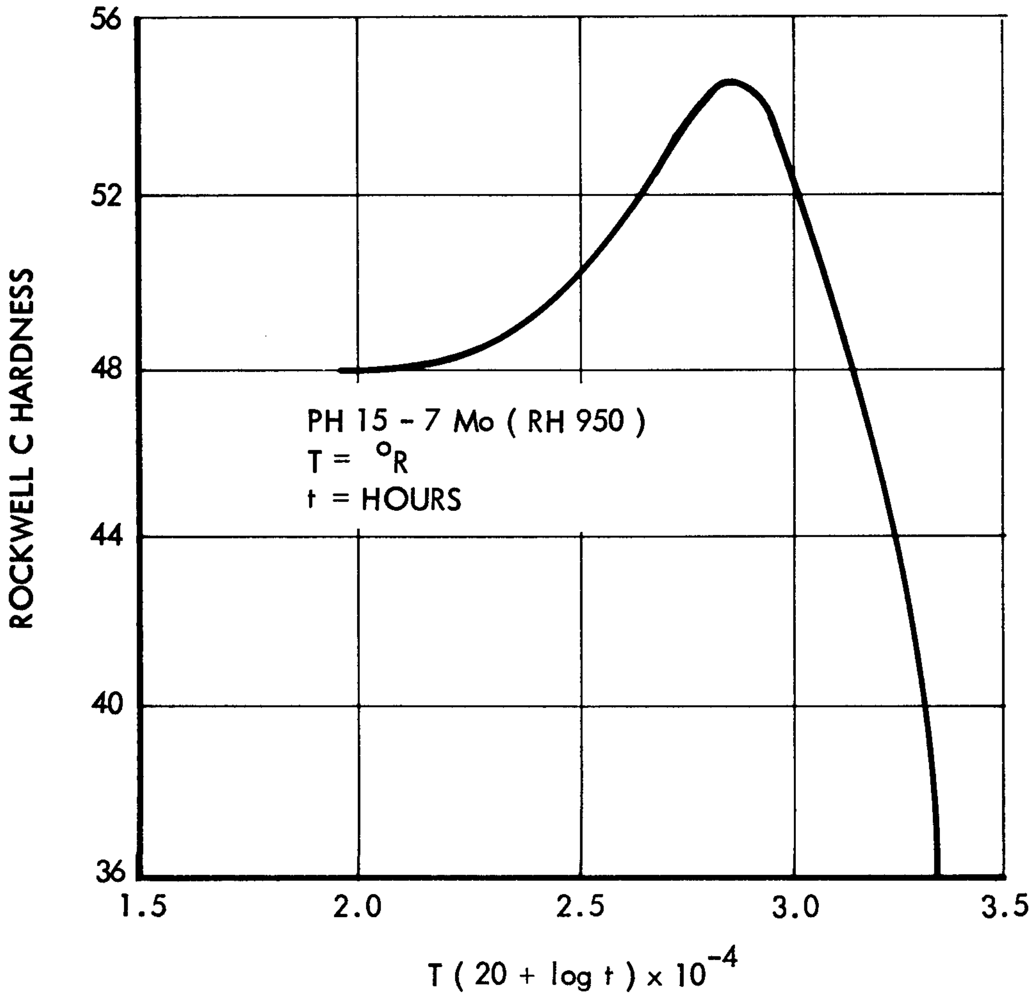


FIGURE 29. MASTER AGING CURVE FOR PH 15-7 Mo (CONDITION RH 950) (3)

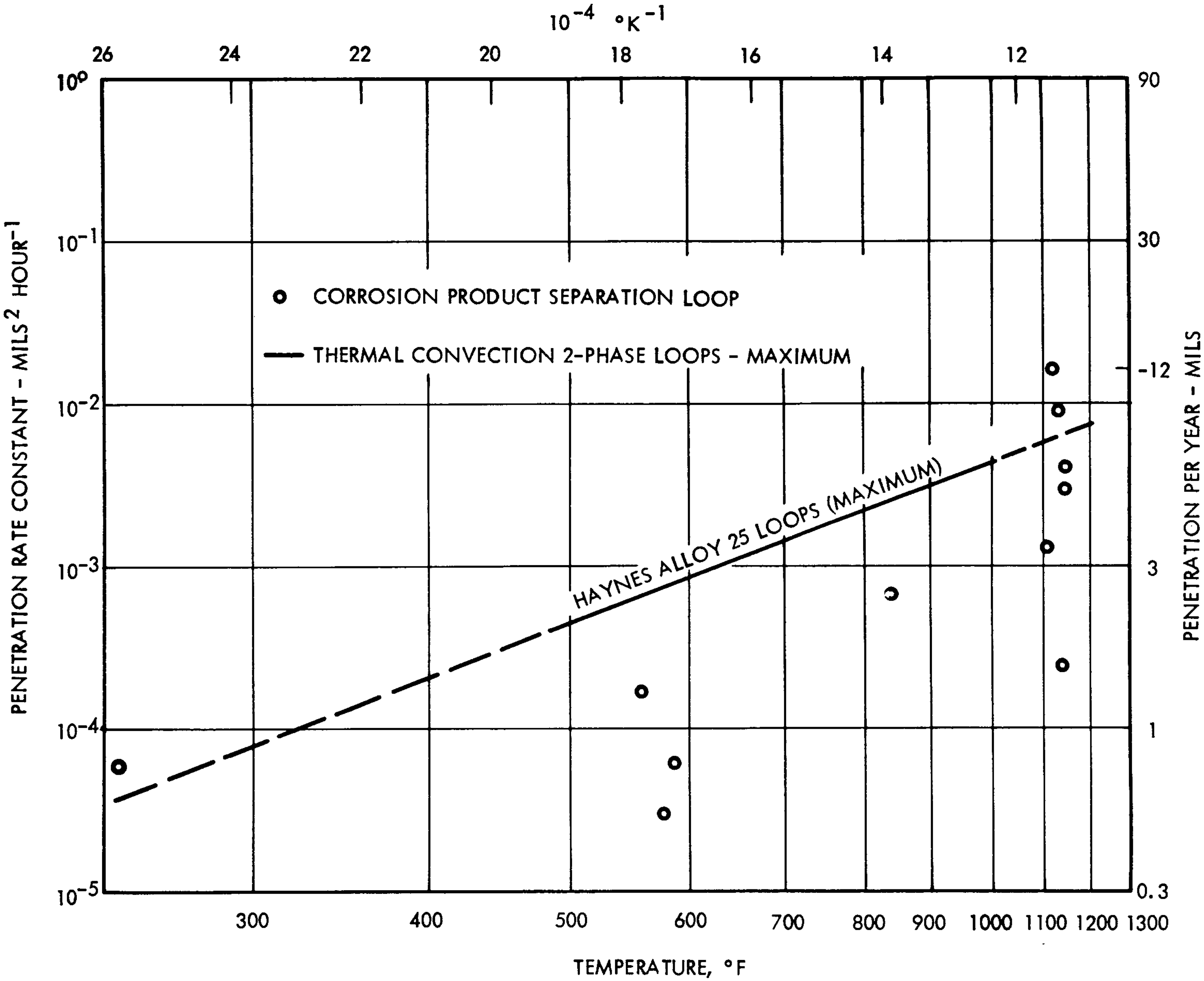


FIGURE 30. COMPARISON OF HAYNES ALLOY NO. 25 MERCURY PENETRATION FROM CORROSION PRODUCT SEPARATION LOOP WITH THERMAL CONVECTION, TWO-PHASE LOOPS (4).

In general, the results indicate that the greatest attack occurred in the low vapor quality regions of the boiling section of the loop and that the attack decreased as the vapor quality increased. Near the outlet of the superheater, where condensation was beginning to take place, deposition was observed. This continued into the region of the orifice and nozzle as the vapor quality continued to decrease. As a result of the increase in liquid content of the vapor, corrosion of the orifice (up to 7.3 mils) and of the nozzle (up to 5.0 mils) occurred. The Nivco 10 and PH15-7Mo erosion specimens also suffered some attack and erosion damage, but this was not severe. Minor attack was observed in the condenser and subcooler.

It is believed that condensation occurred in the orifice-nozzle region on the loop because of the cooling effect of the by-pass line in this area. It will be recalled that, when the throttling valves were removed from the system, the by-pass line was capped and remained in the loop.

Extrapolation of Figure 30 shows that, under the conditions of this loop test, a maximum penetration of 12 mils might be expected in a Haynes alloy No. 25 system operated for a year's duration. The average penetration, however, could be expected to fall in the range of 2 to 6 mils per year.

2. Deposition

Analysis of deposits removed from the loop and separators are presented in Table 6. Mercury analysis is presented in Table 7. Deposit breakdown for the system was as follows: pre-heater and boiler, 45%; vapor corrosion product separator, 26%; superheater, 2%; orifice, 0.1%; nozzle, 24%; condenser, 0; liquid corrosion product separator, 4%; and subcooler, 0. A total of 22.4 grams of leached metal was recovered with an overall composition of 57.8% cobalt, 9.3% chromium, 9.0% nickel, 7.5% iron, and 16.5% tungsten.

The mercury was removed from the loop system progressively as described in the procedure. The mercury contained only a small amount of corrosion products. Examination of Table 7 indicates that most of the corrosion products were found at or near the mercury surface, indicating that the majority of the corrosion products separated from the mercury after shut down. For example, the last samples of mercury removed from the boiler contained the majority of corrosion products (sample Nos. 2 and 3).

TABLE 6

CHEMICAL ANALYSIS OF DEPOSITS FROM LOOP

Sample Location	Average Temp., °F	Total Wt. of Sample Grams	Weight of Hg in Sample Grams	Weight of Corrosion Products in Sample Grams	Percent of Corrosion Products	Composition of Corrosion Products, Weight Percent: Co	Cr	Ni	Fe	Mn	W	Al	Cu	Ti	Zr
Venturi	100	5.3883	5.3640	0.0243	0.11	65.0	17.7	13.0	3.2	0.2	1.0	0	0	0	0
Pre-Heater Inlet (Section 12)	838-925	19.8390	19.3250	0.5140	2.29	83.9	9.3	0.8	5.5	<0.01	0.4	0	0	0	0
Pre-Heater Mid-Section (Section 11)	925-1077	9.8360	7.0850	2.7510	12.3	44.3	2.6	1.3	2.0	<0.01	50.0	0	0	0	0
Pre-Heater Outlet (Section 10)	1077-1106	22.6117	16.4467	6.1650	27.6	43.2	3.2	1.5	1.6	<0.01	50.7	0	0	0	0
Boiler Inlet (Section 8)	1106-1140	0.0590	0.0244	0.0346	0.15	52.7	16.4	5.7	10.0	0.2	15.0	0	0	0	0
Boiler Mid-Section (Section 7)	1124-1140	1.9160	1.3860	0.5300	2.36	54.4	16.0	3.7	4.8	<0.02	21.0	0	0	0	0
Boiler Outlet (Section 6)	1124-1137	1.4150	1.3220	0.0930	0.42	42.1	17.0	8.9	8.1	0.2	23.6	0	0	0	0
Superheater Inlet (Section 5)	1166-1212	0.4890	0.4350	0.0540	0.24	46.0	13.1	17.5	7.7	0.3	15.3	0	0	0	0
Vapor Corrosion Product Separator Inlet	1188	20.6160	16.8720	3.7440	16.7	60.3	19.8	9.7	3.8	<0.03	6.3	0	0	0	0
Vapor Corrosion Product Separator-Columbium Turnings (Alloy D-36)	1188-1191	~40	~38	2.0852	9.34	69.0	3.6	25.8	1.5	<0.05	0	0	0	0	0
Superheater Outlet (Section 1)	1305-1373	0.3540	Nil	0.3540	1.58	50.6	29.0	16.1	3.5	<0.02	0.8	0	0	0	0
Nozzle Outlet	862	6.1264	0.7915	5.3349	23.8	54.0	20.2	10.9	3.7	0.1	11.0	0	0	0	0
Liquid Corrosion Product Separator Columbium Turnings (Alloy D-36)	397-527	~10	~10	0.4389	1.96	10.4	3.2	7.5	78.8	0.1	0	0	0	0	0
Iron Wool (Bottom)		~250	~250	0.0160	0.72	23.8	40.1	30.6	0	5.6	0	0	0	0	0
Magnets (Pole Pieces)		0.2520	Nil	0.2520	1.13	16.1	1.5	13.7	68.2	0.3	0.3	0	0	0	0
Iron Wool (Top)		~125	~125	0.0310	0.14	<0.01	56.2	21.0	0	0.6	22.3	0	0	0	0
Orifice	796	0.0215	Nil	0.0215	0.10	Major	Major	Major	0.5-5	0.5-5	0	0.005-0.05	0.05-0.5	0.005-0.05	<0.01
				22.4434 grams											

Zero indicates that the element was not detected by quantitative analytical techniques.

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TABLE 7

SPECTROGRAPHIC ANALYSIS OF MERCURY DRAINED FROM THE LOOP

Sample Location	Analysis, Weight Percent (Approximate)																	
	Al	B	Ca	Cr	Co	Cb	Cu	Fe	Mg	Mn	Mo	Ni	Na	Si	Ag	Ti	W	Zr
Condenser	-*	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Boiler - Sample No. 1	-	-	.00008	-	-	-	-	.00008	.0008	-	-	-	-	-	-	-	-	-
Boiler - Sample No. 2	-	.0004	.0004	.0044	.0221	-	.0500	.0044	.0044	-	-	.0221	.0044	.0044	.0004	-	.0004	-
Boiler - Sample No. 3	-	-	.00007	.0014	.0360	-	.0072	.0018	.0014	.0072	-	.0144	.0072	.0007	.0004	.0007	.0001	-
Liquid Corrosion Product Separator Sample No. 1	-	-	.0001	-	-	-	-	.0001	.0001	-	-	-	-	-	-	-	-	-
Liquid Corrosion Product Separator Sample No. 2	-	-	.0002	-	-	-	-	.0002	.0002	-	-	.0002	-	-	-	-	-	-
Liquid Corrosion Product Separator Sample No. 3	.0012	.0001	.0012	.0125	.0250	.0125	.0012	.0062	.0025	.0062	.0025	.0062	-	.0062	.0001	.0125	.0012	.0025
Liquid Corrosion Product Separator Sample No. 4	.0076	.0015	.0076	.0152	.0304	.0152	.0030	.0152	.0076	.0076	.0076	.0076	-	.0152	.0002	.0152	.0015	.0030

*Dash signifies nil.

The mercury from the condenser was clean and no corrosion products were detected spectrographically. On the other hand, the mercury from the pre-heater and boiler contained, in decreasing order, the alloying elements cobalt, nickel, iron, chromium, titanium, and tungsten, Table 7. The mercury from the liquid corrosion product separator contained the alloying elements (in decreasing order) cobalt, chromium and iron, nickel, and finally tungsten. The presence of columbium and titanium in the mercury drained from the liquid corrosion product separator indicates that some dissolution of the columbium alloy (D-36) getter in this separator had occurred. Although visual examination of the Alnico 5 magnets in this separator revealed no degradation, it is possible that the aluminum contents listed in Table 7 are a reflection of some attack of the magnets by mercury.

The deposit in the pre-heater section of the loop (Sections 10 through 12) was high in tungsten and low in chromium and nickel, Table 6. The largest penetrations occurred in this region and the deposit composition, therefore, probably reflects the residual corrosion layer composition (which would be high in tungsten and low in nickel and chromium), since some of the corrosion layer was undoubtedly removed when the deposit was scraped from the tubing. There was an overall tendency for the cobalt, nickel, and chromium percentages to increase from the boiler through the superheater and finally through the liquid corrosion product separator. On the other hand, the tungsten percentage had a tendency to decrease.

The columbium alloy turnings in the vapor separator removed primarily cobalt and secondly nickel. The columbium turnings in the liquid separator removed primarily iron. Since the columbium was located in the magnet cavity, it is possible that the presence of a magnetic field was responsible for the iron removal. The iron wool removed chromium, nickel, and cobalt. The magnet removed principally iron and secondly cobalt and nickel.

The orifice deposit was essentially Haynes alloy No. 25 with the exception of tungsten. The nozzle outlet deposit was also essentially Haynes alloy No. 25 and did contain tungsten. Since the nozzle was made of Nivco 10, an alloy free of tungsten and chromium, these deposits were obviously mass-transported into this area from Haynes alloy No. 25 dissolution elsewhere in the system. Since this large mass of deposit could not have been solely generated in the superheater, then it must have been the result of mass-transport of material from the pre-heater and boiler where relatively large amounts of corrosion products were being produced.

The small quantity of amalgam in the venturi was the result of condenser and subcooler dissolution and possible carry-over from the boiler. The venturi was at 100°F, the lowest temperature in the system.

Corrosion results show that the greatest generation of corrosion products occurs in the boiler, the area contacted by the hottest liquid mercury. Only small amounts of corrosion products are generated in the low temperature liquid-contacted condenser. The corrosion products tend to accumulate in the boiler and, hence, they tend to be mass-transported through the vapor regions of the system. This is the reason why the vapor corrosion product separator contained substantially more corrosion products than the liquid corrosion product separator after the condenser. In addition, the temperature of the liquid separator, 527 to 397°F, was low and gettering action as a result was moderately light. However, the action of the liquid separator was sufficient to prevent deposition in the subcooler since this area was devoid of deposits.

The vapor separator inlet contained 17 percent of the corrosion products. This separation was the result of baffling action which removed some of the liquid mercury droplets from the vapor stream. An additional nine percent of the corrosion products were contained by the columbium alloy turnings in the vapor separator. The efficiency of the vapor separator may be approximated by considering the quantity of corrosion products found downstream of the separator, since the separator can be expected to collect only those corrosion products that pass through it. On this basis, the separator collected a total of 5.8292 grams of corrosion products, while a total of 5.7104 grams were found downstream of the separator in the superheater outlet, orifice, and nozzle (see Table 6). The separator was thus approximately 50.6 percent efficient in removing corrosion products carried over from the boiler.

CONCLUSIONS

On the basis of the specific test conditions reported, the following conclusions are made:

1. Operation of a forced circulation, Haynes alloy No. 25, mercury system for up to 5200 hours has been demonstrated to be feasible.

2. Corrosion data for this system agree favorably with data for two-phase, thermal convection loops.

3. The greatest penetrations occurred in the low vapor quality regions of the boiling section of the loop, one isolated portion of the superheater inlet, and in the orifice and nozzle where condensation was occurring.

4. The condenser and subcooler sections of the loop suffered negligible attack, as did the dry portions of the superheater.

5. Welds were unattacked in all cases except one where a crack was observed in the weld. The attack ($<$ 1 mil) was minor.

6. Extrapolation of the data shows that a maximum penetration of 12 mils might be expected in this system if operated for a year's duration. The average penetration, however, could be expected to be much less (two to six mils).

7. The greatest quantity of deposition was observed in the pre-heater section and in the orifice and nozzle where condensation was occurring.

8. A deposit was observed in the orifice after shut-down which was blocking 99.2 percent of the flow passage. It is believed that the majority of the deposit was formed during the shut-down operation.

9. The action of the liquid corrosion product separator prevented deposition in the subcooler section.

10. The vapor corrosion product separator was approximately 51 percent efficient in removing corrosion products carried over from the boiler.

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2. Nejedlik, J. F., "The SNAP II Power Conversion System Topical Report No. 14. Mercury Materials Evaluation and Selection." NAA-SR-6306. April 15, 1962.

3. Nejedlik, J. F., "Evaluation of Sunflower CSU I-3A Turboalternator Components after Six Months of Operation." TRW Technical Memorandum TM 3893. January 9, 1964.

4. Nejedlik, J. F., "Analysis of Sunflower CSUI-3 Turbine Inlet Housing Corrosion and Deposits." TRW Technical Memorandum TM 3613-67. January 18, 1963.

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APPENDIX

ELECTRON BEAM MICROANALYSIS RESULTS

An electron beam microanalysis was performed by Advanced Metals Research Corporation on three of the specimens taken from the loop. The specimens examined were the pre-heater outlet, the condenser outlet, and the nozzle throat. The analysis of the pre-heater and condenser specimens included the elements comprising the columbium alloy getter used in the corrosion product separators (columbium, titanium, and zirconium) to determine if mass transfer of these elements had occurred.

As seen in Figures 31 through 34, the electron beam microanalysis on the pre-heater outlet specimen (see Figure 20b) revealed a leached layer with an abrupt concentration discontinuity approximately 40 to 45 microns from the surface. The corrosion layer was found to be enriched in tungsten and depleted in nickel, chromium, and cobalt. Zirconium was found in the matrix as tungsten-zirconium carbides and comprised less than 0.05 percent. The zirconium concentration rose to 0.05 to 0.08 percent in the corrosion layer; however, the background intensity also rose as a result of the increased tungsten content. The titanium and columbium concentrations were consistently less than 0.05 percent.

The condenser outlet specimen (see Figure 22d) revealed no visible evidence of a surface chemistry change. However, the concentration distributions (Figures 35 through 38) showed the presence of a slowly varying gradient to a depth of 25 to 30 microns. Cobalt, nickel, and tungsten showed an increase at the I.D. surface of the specimen, while chromium was found to be depleted. A small metallic chip, partially buried in the mount material, was included in the traverse. This chip was the only one noted in the area selected for analysis and is considered insignificant. Except for some zirconium in the matrix tungsten-zirconium carbides, the zirconium, titanium, and columbium concentrations were consistently less than 0.05 percent.

Examination of the nozzle throat specimen (see Figure 27b) revealed a surface zone of intergranular precipitation extending to a depth of approximately 70 microns. A series of large gray precipitates near the base metal-end of the surface zone were also noted. The gray phases and oxides were found to be zirconium oxides that appeared to also contain some nickel in solution. These phases were identified as oxides since they yielded a visible fluorescence when struck by the electron beam. This fluorescence is typical of the presence of combined oxygen. The composition of the metal matrix surrounding the oxides was found to be essentially the same as the base metal. The concentration distributions for chromium and tungsten were consistent at less than 0.05 percent.

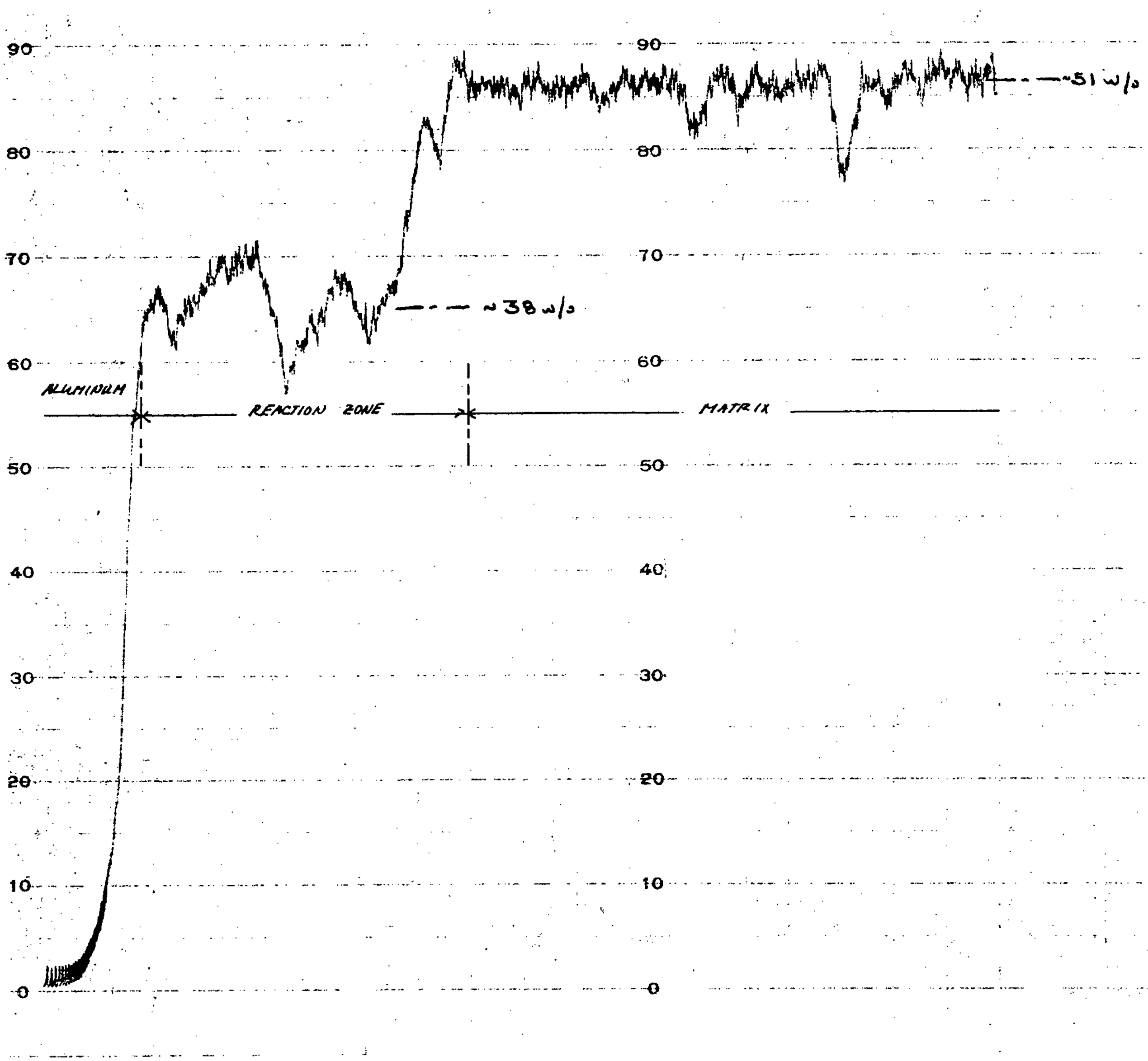

Figure 31. Distribution of Cobalt in the Pre-Heater Outlet Specimen.

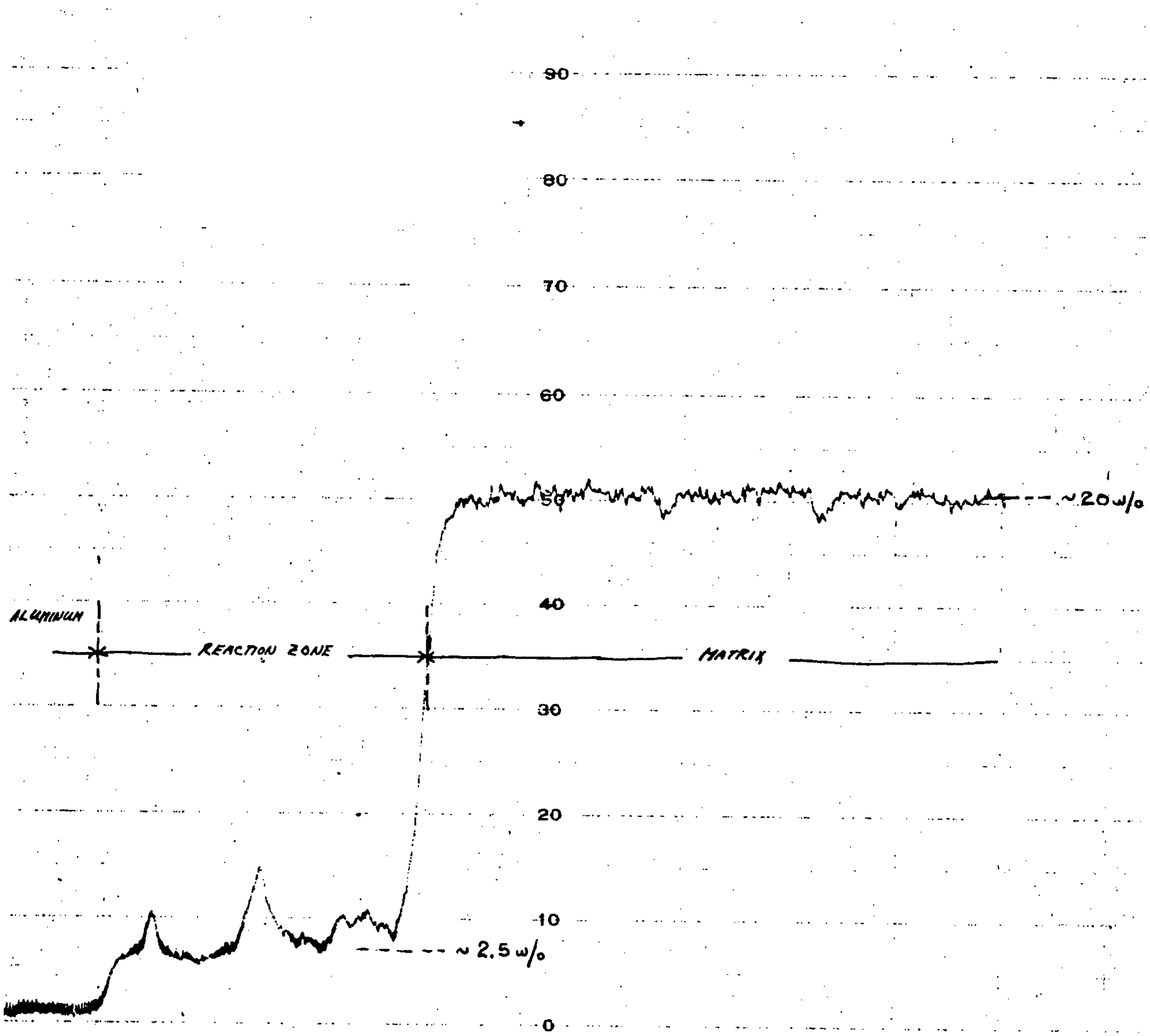

Figure 32. Distribution of Chromium in the Pre-Heater Outlet Specimen.

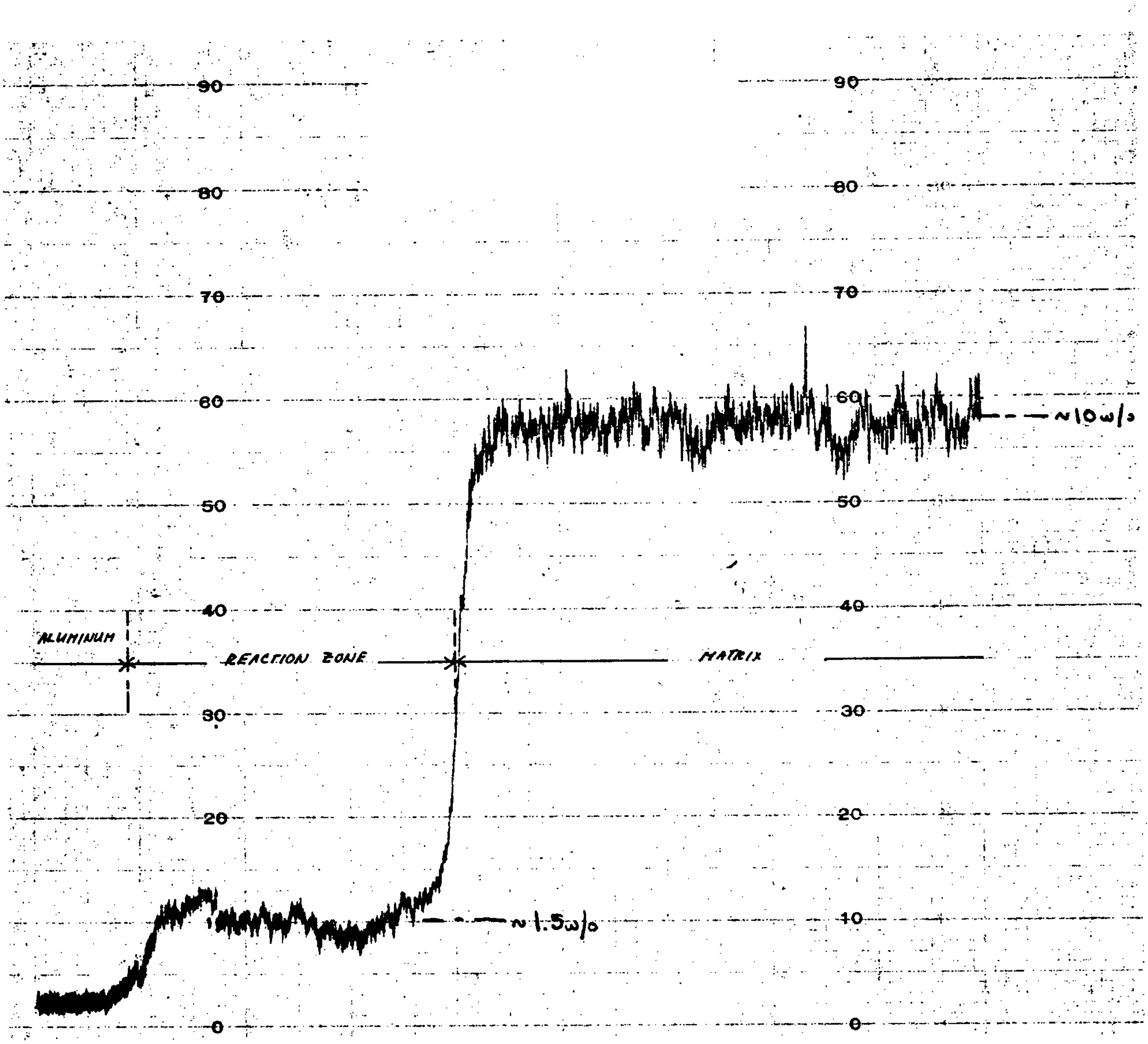

Figure 33. Distribution of Nickel in the Pre-Heater Outlet Specimen.

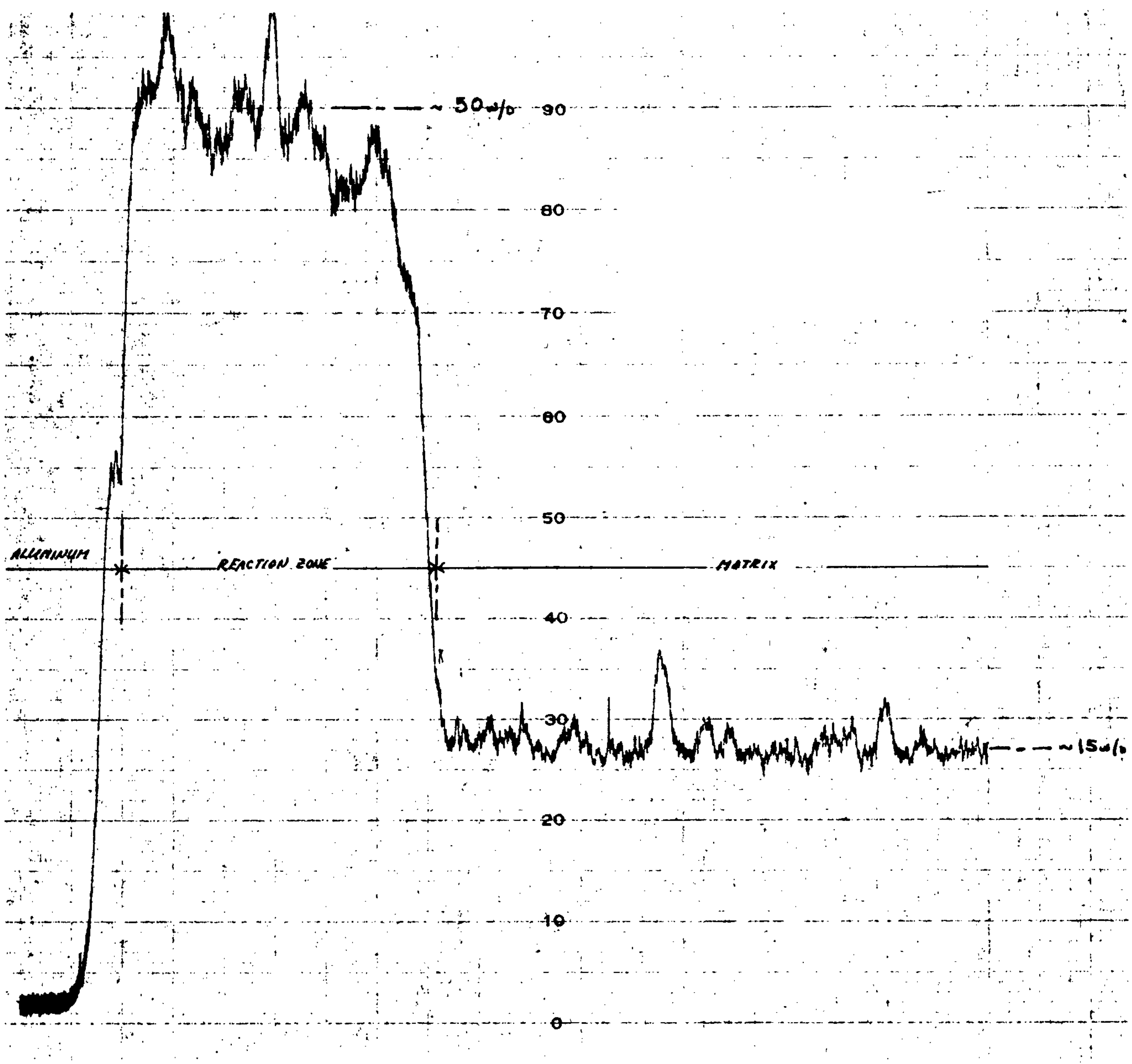

Figure 34. Distribution of Tungsten in the Pre-Heater Outlet Specimen.

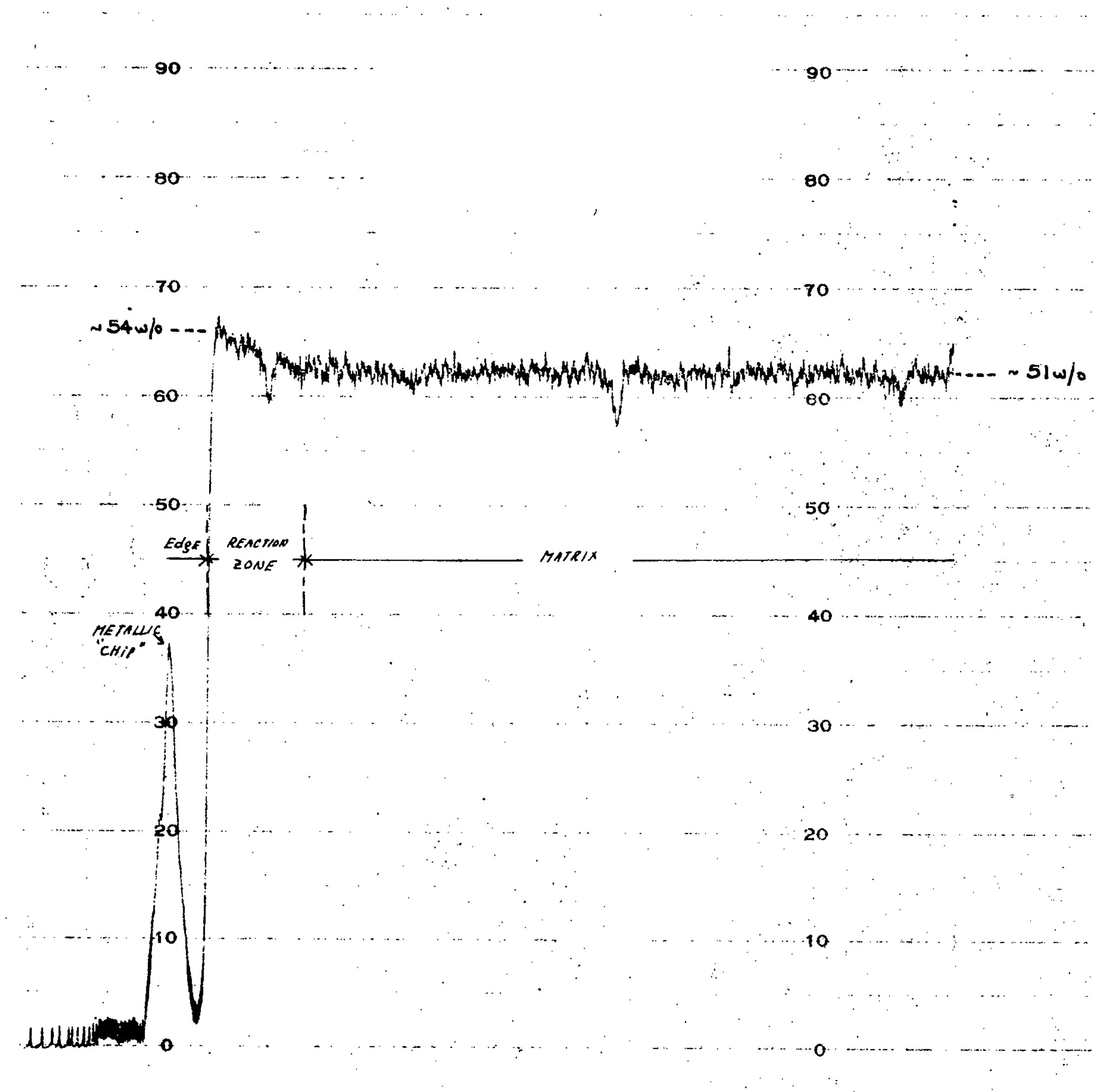

Figure 35. Distribution of Cobalt in the Condenser Outlet Specimen.

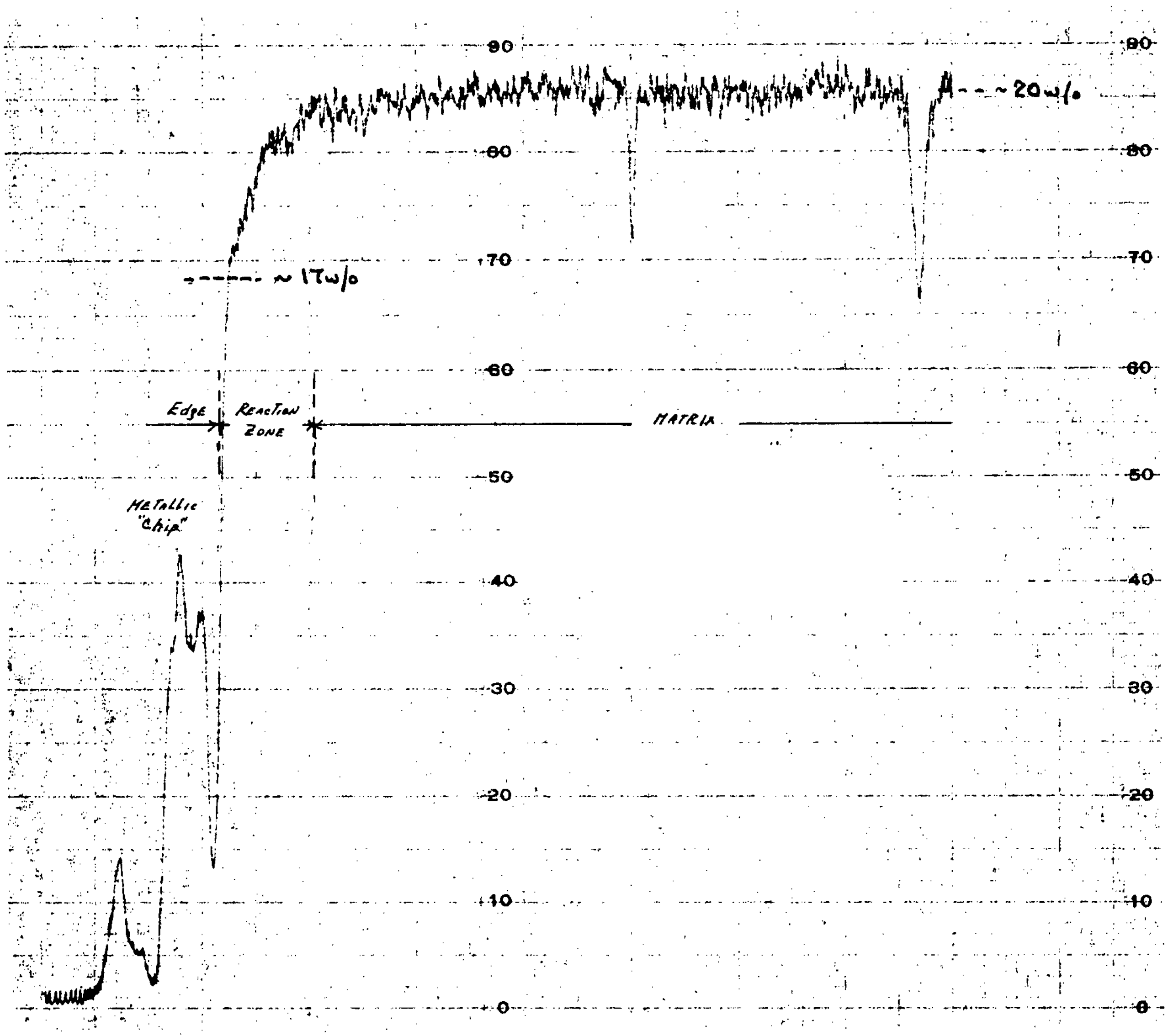

Figure 36. Distribution of Chromium in the Condenser Outlet Specimen.

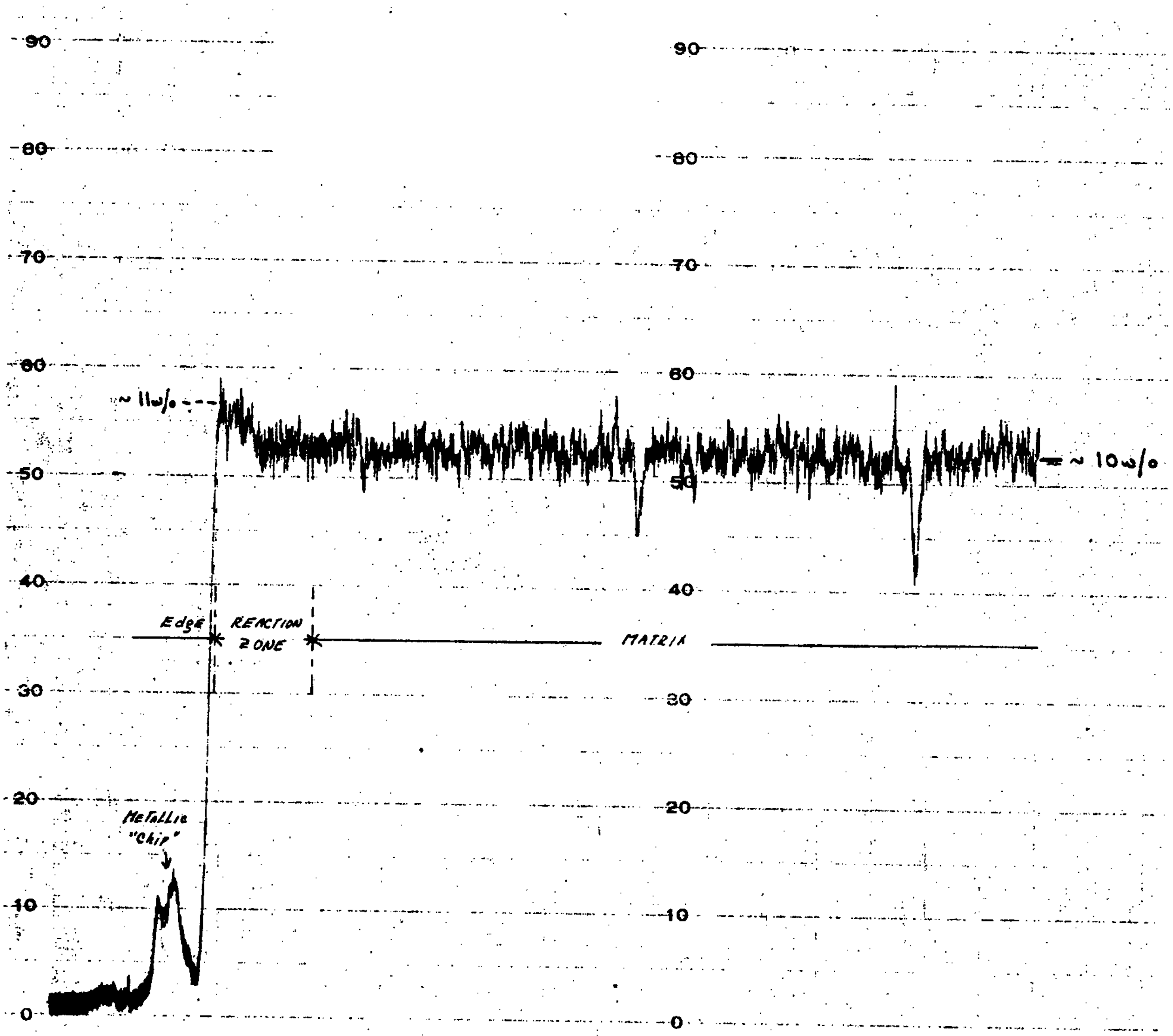

Figure 37. Distribution of Nickel in the Condenser Outlet Specimen.

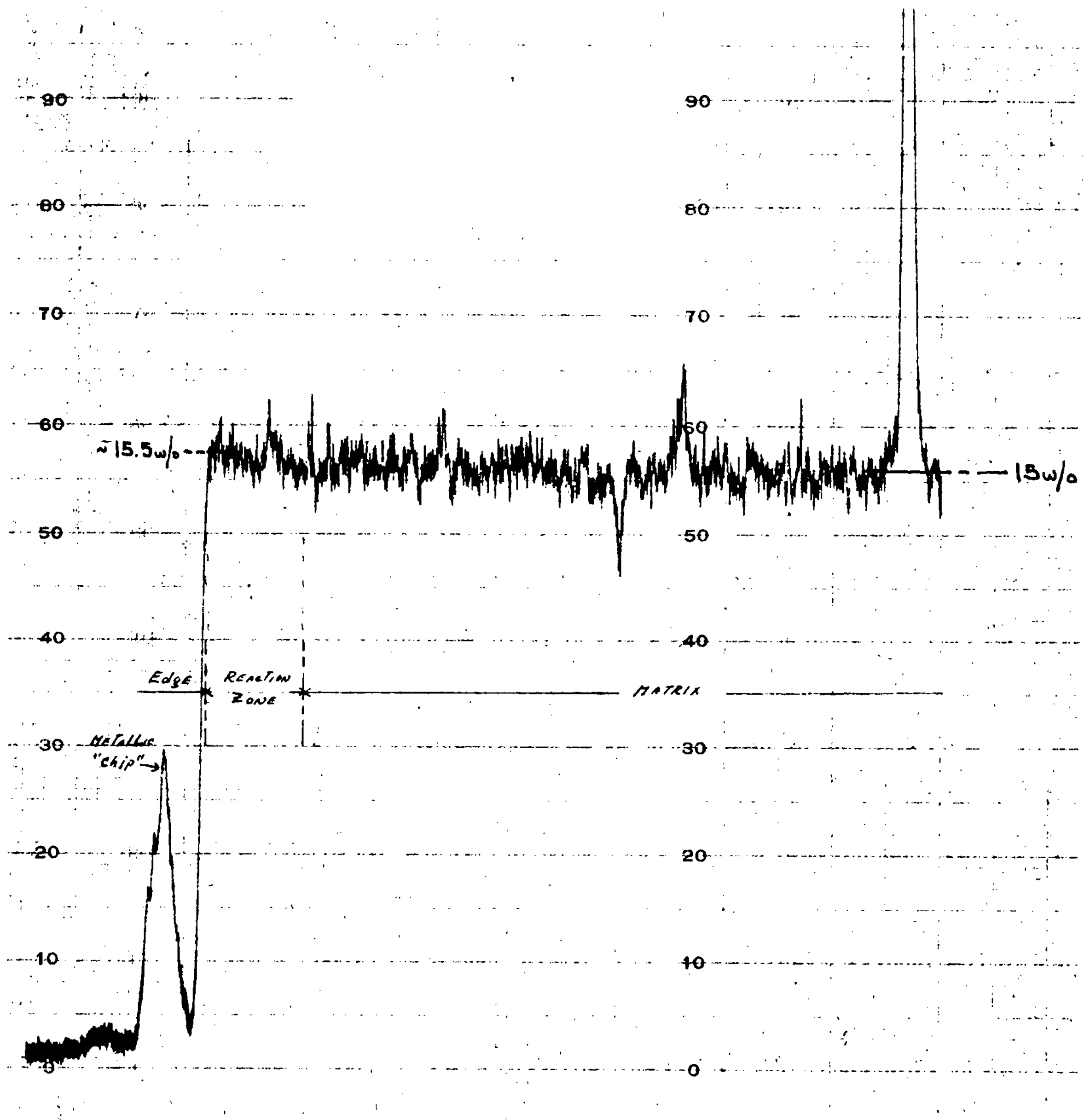

Figure 38. Distribution of Tungsten in the Condenser Outlet Specimen.

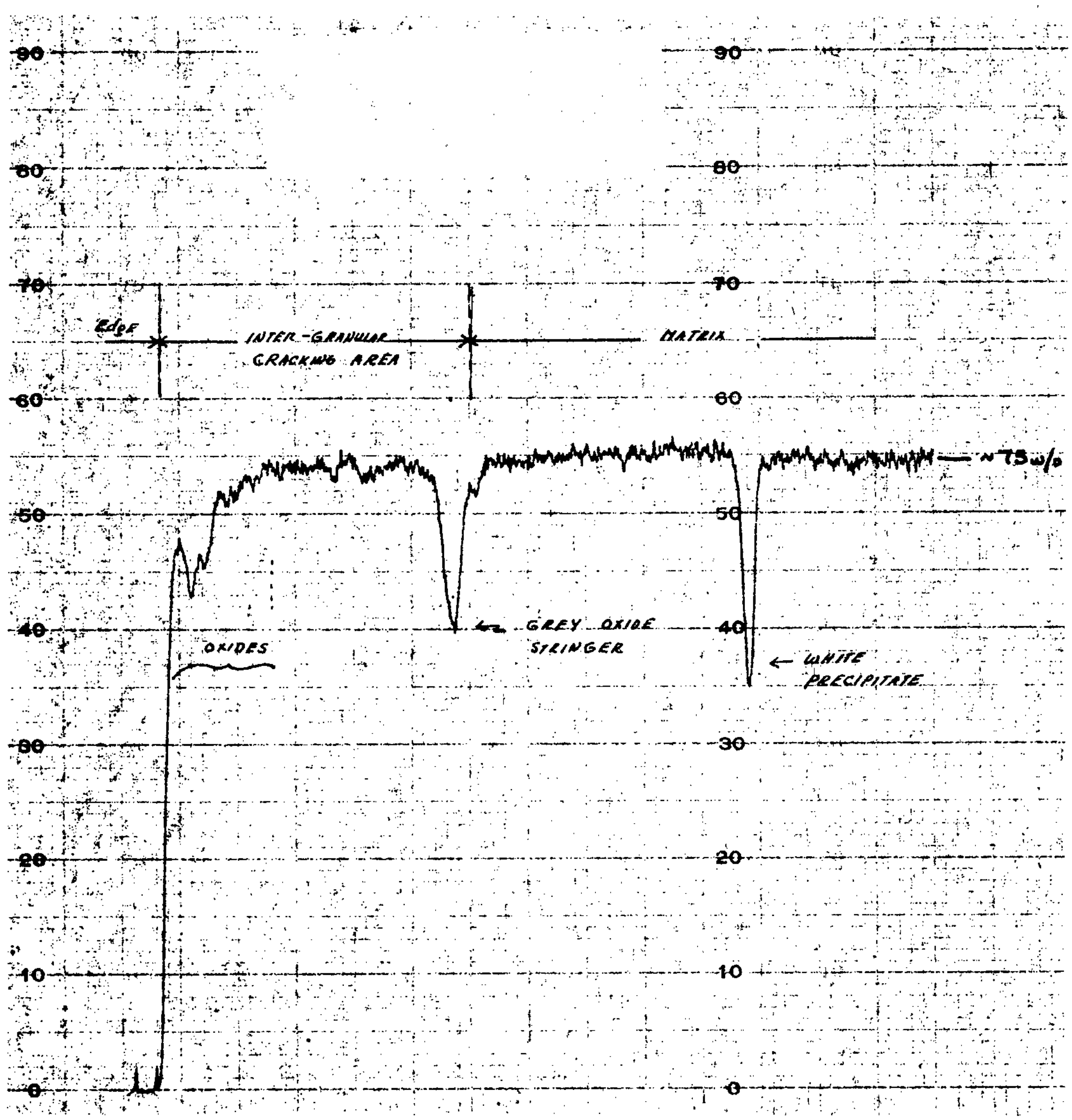

Figure 39. Distribution of Cobalt in the Nozzle Throat Specimen.

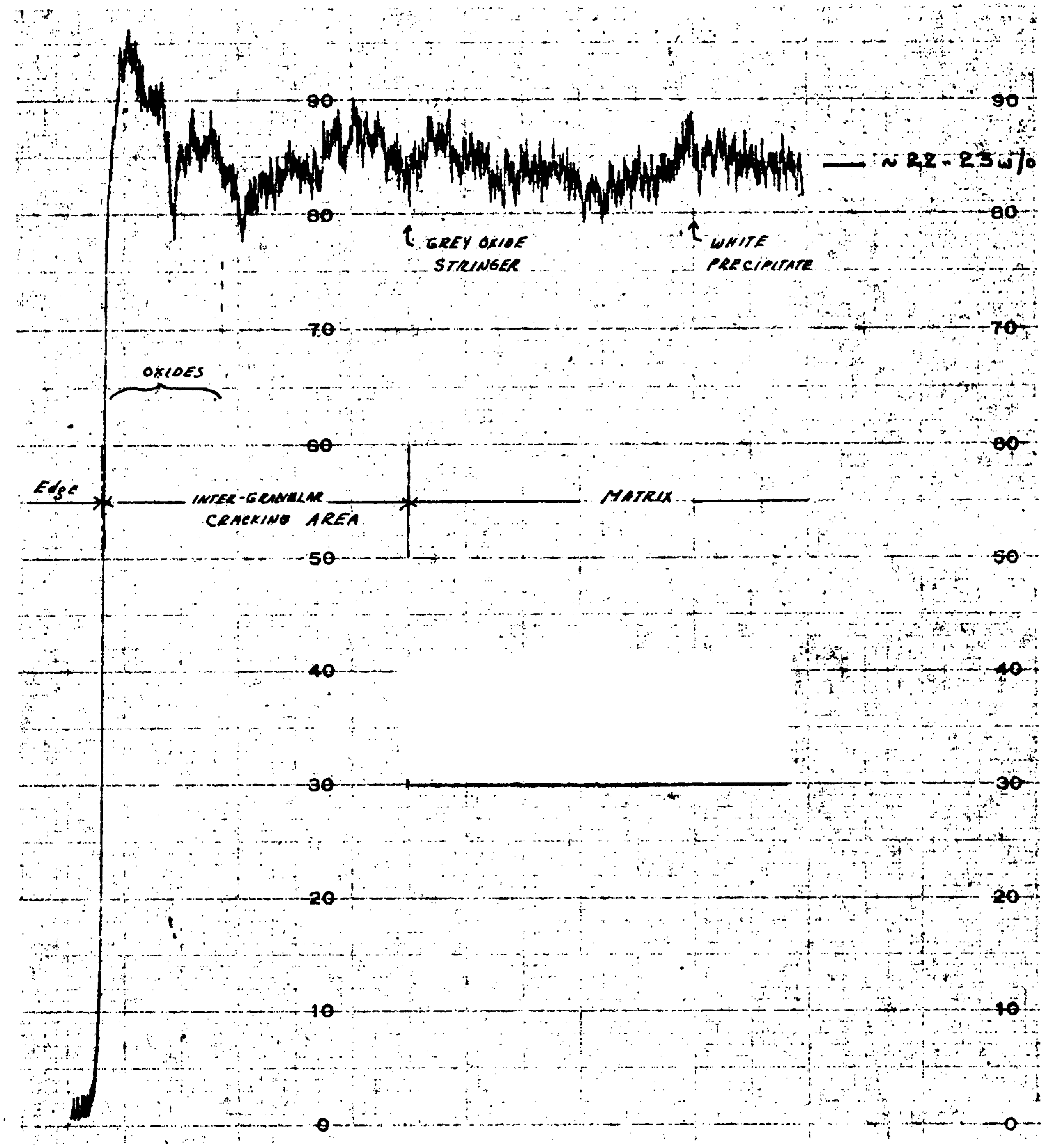

Figure 40. Distribution of Nickel in the Nozzle Throat Specimen.

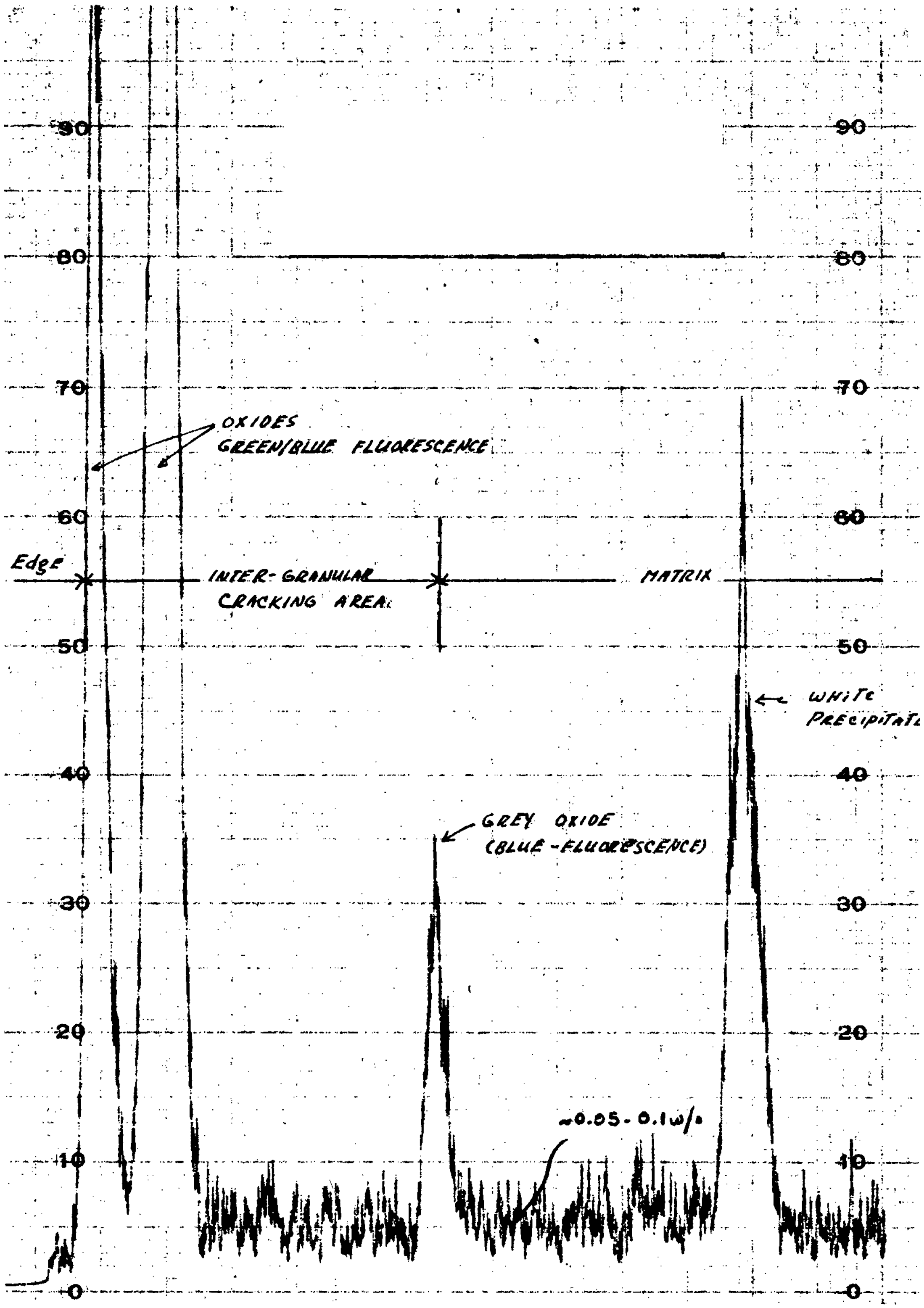

Figure 41. Distribution of Zirconium in the Nozzle Throat Specimen.

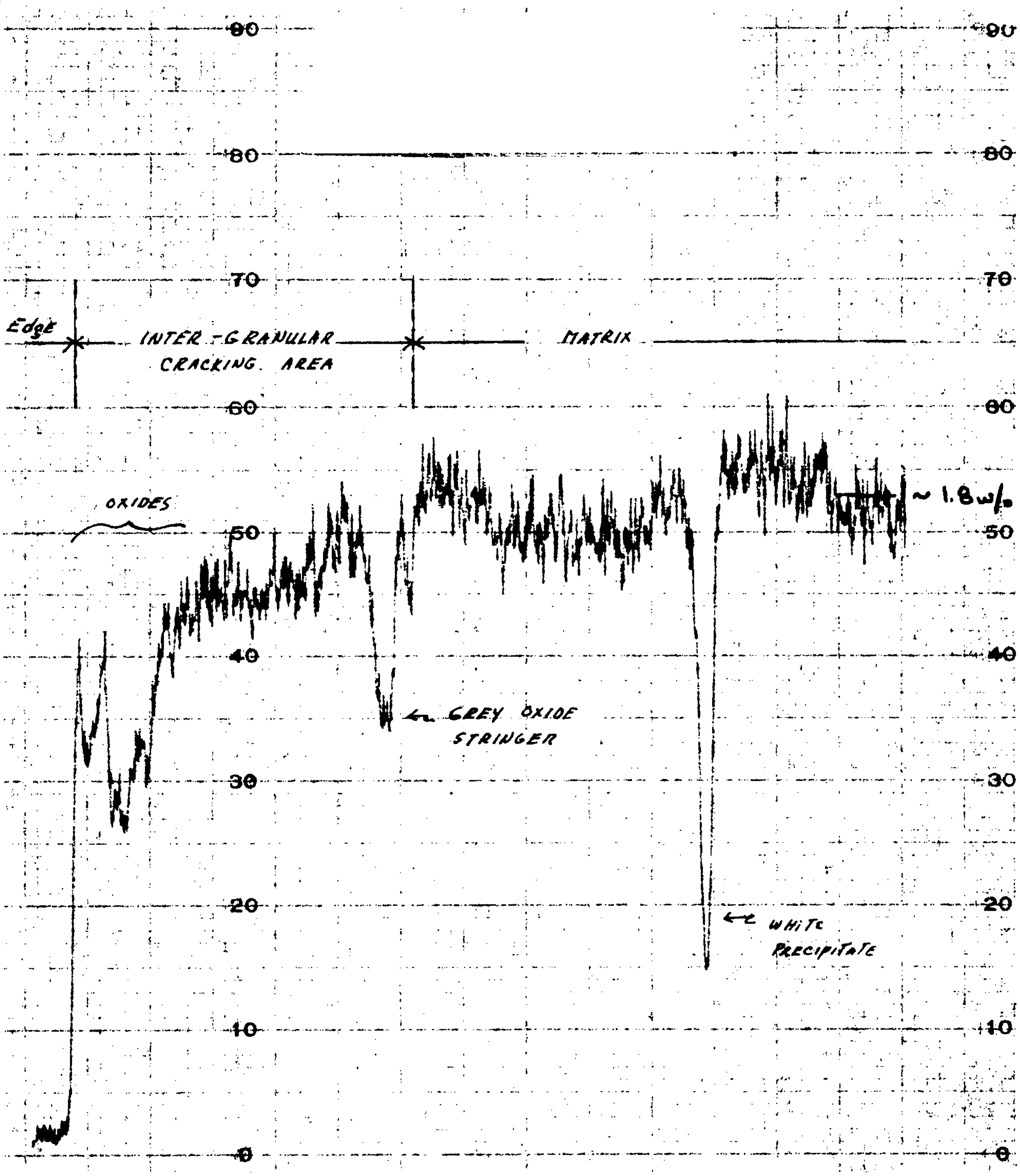

Figure 42. Distribution of Titanium in the Nozzle Throat Specimen.

NASA-Langley, 1965

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